ISBS
Institut für Straßenwesen
TU Braunschweig

Asphalt Binder Testing using 4 mm parallel plate geometry of Dynamic Shear Rheometer

Interlaboratory Study

including test results of 20 participating laboratories from Belgium, Czech Republic, Denmark, Finland, France, Germany, Italy, Lithuania, and Poland

Final Report by

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https://doi.org/10.24355/dbbs.084-202111031047-0

Table of Contents

https://doi.org/10.24355/dbbs.084-202111031047-0

1 Motivation

Today, there is no test standard in Europe for low temperature testing of asphalt binders with the Dynamic Shear Rheometer (DSR), even though the 4 mm parallel plate geometry is noted in the draft standard prEN 14770. Currently, there are few laboratories using this geometry and only limited information is available on sample geometry, sample preparation, sample conditioning, test procedure and test repeatability.

However, DSR testing by use of the 4 mm parallel plate geometry is gaining more and more interest in the asphalt community for assessing low temperature properties of asphalt binders. Therefore, this interlaboratory study was initiated and sponsored by the Braunschweig Pavement Engineering Centre (ISBS) at Technische Universität Braunschweig in 2019, in order to compare different test protocols currently in use, specifically in view of repeatability and reproducibility. In this report, information on different test methods is provided, and the respective test results are statistically analyzed and compared.

ISBS thanks all laboratories for participating in this interlaboratory study.

2 Materials

In this interlaboratory study, penetration graded asphalt binders were used, i. e. plain asphalt binder of the type 50/70 and polymer modified asphalt binder of the type 25/55-55 A (according to EN 12591, 2009 and EN 14023, 2010, respectively) (see Table 1).

Table 1: Asphalt binders of the penetration graded types 50/70 and 25/55-55 A

Test Method		50/70	25/55-55 A
Softening Point Ring-and-Ball (EN 1426, 2015)	[°C]	51.3	58.0
Needle Penetration (EN 1427,2015)	[1/10mm]	64	46

The batches for both asphalt binders consisted of 40 buckets of about 10kg, provided by the manufacturer in November 2017.

For sample production, 10kg of each binder was heated in oven and homogenized in accordance with EN 12594 (2012), and then separated into 25 containers of 350ml per binder. Sampling took place on August 27th, 2019. On August 28th one container of binder 50/70 (approx. 350ml) and one container of binder 25/55-55 A (approx. 350ml), together with safety data sheets was sent to each participant of the interlaboratory study. No sample was reported damaged or missing.

3 Instructions

All participants received instructions for testing per email from August 29th.

A temperature-frequency sweep (T-f-sweep) test was performed on both binders, with two test repetitions. Test temperatures were -30 (if possible), -20, -10 and 0°C. Test frequencies were 0.1; 1.0; 1.59 and 10Hz.

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The participants applied their usual procedure when using 4 mm geometry. No detailed instructions on sample preparation and material testing were provided. Sample preparation, sample conditioning, oscillation amplitude was individually chosen by each participant.

An Excel file was provided to collect information on the device used, on sample geometry, sample preparation, specimen conditioning, test conditioning and test results (phase angle δ and complex shear modulus G*). In addition, participants had the possibility to comment their specific methods.

4 Registration and submission

20 laboratories participated in this interlaboratory study, covering Belgium, Czech Republic, Denmark, Finland, France, Germany, Italy, Lithuania, and Poland. One participant provided three separate results with different testing procedures. One participant was not able to submit results for technical reasons. A total number of 22 test protocols was received, from September 24th, 2019 to January 13th, 2020.

5 Analysis

Anonymous laboratory numbers are assigned to all test results. Any participant knew his specific number only.

General information on test equipment and test procedure is summarized in Section 6.

Based on the resulting values for complex shear modulus and phase angle of all data sets, the following parameters were calculated (cp. Section 6):

- *mean value* of the two individual test results,
- *deviation* of individual values from the mean value in percent,
- *general mean* of all test results from all participants, except those with *z score* ≥ 3.0,
- *lab deviation* of mean value from each participant from general mean of all test results in percent,
- *z score*; the z score was chosen as the statistical criterion. The z score for a result of a proficiency test is calculated from: $z_i = \left(x_i - x_{pt}\right)/\sigma_{pt}$ according to Section 9.4 of Standard ISO 13528, where x_i is the assigned value, x_{pt} is the robust average of the laboratory results before statistical exclusion, but after analytical exclusions, and σ_{pt} is the standard deviation for the proficiency assessment. The conventional interpretation of z scores is as follows (see ISO/IEC 17043,2010):
    - result that gives $|z| \leq 2{,}0$ is considered satisfactory,
    - result that gives $2{,}0 < |z| < 3{,}0$ is considered to be a warning signal,
    - result giving $|z| \geq 3{,}0$ is considered unsatisfactory (or intervention signal).

Detailed statistical analysis is provided for a test frequency of 1.59Hz only, results for frequencies of 0.1; 1.0 and 10Hz are presented graphically in Section 6.

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The *deviation* is used to evaluate the repeatability of test results for each participant. The *lab deviation* is used to evaluate the overall reproducibility of the test method. It should be noted that the test results are not obtained under reproducibility conditions, as different methods in terms of sample preparation, sample geometry and sample conditioning were used.

https://doi.org/10.24355/dbbs.084-202111031047-0

6 Test protocols and test results

6.1 Dynamic Shear Rheometer used in this study

Laboratory	Brand of DSR	Model of DSR	Year of purchase	DSR control software
1	Discovery Series	HR-3	2015	Trios
2	Anton Paar	EC Twist 502	2016	RheoCompass 1.23
3	Anton Paar	MCR 102	2013	RheoPlus
4	Anton Paar	SmartPave102	2016	RheoCompass 1.24
5	Malvern Panalytical	DSR	2018	rSpace 1.75
6	Malvern Panalytical	Kinexus Pro	2012	rSpace 1.72
7	Anton Paar	MCR-101	2008	RheoPlus 2.81
8	Anton Paar	MCR 502	2014	RrheoCompass 1.20
9	Malvern Panalytical	Kinexus Pro Plus	2014	rSpace 1.72
10	Anton Paar	MCR 302	2016	RheoCompass 1.24
11	Anton Paar	MCR 302	2019	RheoCompass 1.24
12	Anton Paar	MCR 302	2016	RheoCompass
13	Malvern Panalytical	Ultra+	-	rSpace 1.75
14	Anton Paar	MCR 302	2012	RheoPlus 3.61
15	Anton Paar	MCR 502	2016	RheoCompass 1.23
16	Anton Paar	MCR 302	2014	RheoCompass
17	Malvern Panalytical	Kinexus DSR+	2015	rSpace 1.75
18	Anton Paar	SmartPave 102	2019	RheoCompass 1.24
19	Malvern Panalytical	Kinexus Pro+; DSR+	2017	rSpace 1.75
20	Malvern Panalytical	Kinexus Pro+; DSR+	2017	rSpace 1.75
21	Malvern Panalytical	Kinexus Pro+; DSR+	2017	rSpace 1.75
22	Anton Paar	MCR Smart Pave 102	2018	RheoCompass 1.24

Laboratory	Temperature control system	Model of temperature control system
1	Liquid Nitrogen	ETC Oven
2	Peltier	H-PTD 200
3	Peltier	H-PTD-200
4	Peltier	P-PTD200/56/I+H-PTD200
5	Peltier	-
6	Peltier	KNX2007
7	Peltier	TU1=TEK150PA-CF (this is under Peltier) +H-PTD200- (this is the upper Peltier-was acquired 2018)
8	Peltier	H-PTD 200+P-PTD 200
9	Peltier	-
10	Peltier	H-PTD 200, P-PTD 200/56/I
11	Peltier	H-PTD 200
12	-	H-PTD200 + P-PTD200
13	Peltier	Active Peltier
14	Peltier	C-PTD200+H-PTD200
15	Convection	CTD 180
16	Peltier	H-PTD200
17	Peltier	-
18	Peltier	P-PTD200 + H-PTD200
19	Peltier	KNX2007
20	Peltier	KNX2007
21	Peltier	KNX2007
22	Peltier	P-PTD200+H-PTD120

https://doi.org/10.24355/dbbs.084-202111031047-0

laboratory	Is an external heat exchanger used for counter cooling?	If YES, brand and model	If YES, liquid used for counter cooling	If YES, is the external heat exchanger controlled by the DSR?	If YES, is the external heat exchanger controlled manually?	If YES, counter cooling temperature
1	NO	-	-	-	-	-
2	YES	Julabo FP50	Thermal G	YES	NO	-
3	YES	Julabo FE32 HL	Antifreeze	YES	No but Yes we can	-
4	YES	Julabo FP50	Glycol	NO	YES	8
5	YES	Julabo CF41	Fridex G48	YES	NO	-
6	YES	Julabo CF41	Fridex	YES	NO	-
7	YES	Minichiller Petroplus	Water-Glycerol 50/50	NO	YES	-10
8	YES	Viscotherm VT2	Antifreeze	YES	NO	-
9	YES	Julabo CF41	-	YES	NO	-
10	YES	Viscotherm VT2	Antifreeze	YES	YES	-10
11	YES	Viscotherm VT2	50% Water- 50% Glycerol	YES	YES	-10
12	YES	Viscotherm VT2	Water + 15% Glycol	YES	NO	-
13	YES	Julabo CF41	Thermal G	YES	NO	-
14	YES	ViscoTherm VT2	Ethylene Glycol	NO	YES	5
15	YES	Julabo FP50	Ethanol 96%	YES	NO	-
16	YES	Julabo FP50	Water + Glycol	YES	-	-
17	YES	Julabo CF41	Antifreeze G12+	YES	NO	-
18	YES	Julabo CF41	Water	NO	YES	5
19	YES	Julabo CF41	Thermal G	YES	NO	-
20	YES	Julabo CF41	Thermal G	YES	NO	-
21	YES	Julabo CF41	Thermal G	YES	NO	-
22	NO	-	-	-	-	-

laboratory	Is the 4mm-geometry serrated?	Is compliance correction applied to the test results?	If YES, compliance of 4mm-geometry used	Do you have any previous experiences with the 4mm-geometry?
	(YES/NO)	(YES/NO)	[rad/Nm]	(YES/NO)
1	NO	NO	-	YES
2	NO	NO	-	YES
3	NO	YES	0.0017	YES
4	NO	YES	0.0017	YES
5	YES	NO	-	NO
6	NO	NO	-	NO
7	NO	YES	0.0203	YES
8	NO	YES	0.016	YES
9	YES	-	-	NO
10	NO	YES	0.0224	YES
11	NO	NO	-	NO
12	NO	-	-	NO
13	YES	YES	0.01262	YES
14	NO	YES	0.028	YES
15	NO	NO	-	NO
16	NO	YES (sample 2 field)	0.023	YES
17	NO	NO	-	YES
18	YES	YES	0.016	NO
19	YES	YES	-	-
20	YES	YES	-	YES
21	YES	YES	0.0014	YES
22	NO	NO	-	YES

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Comments

lab. 1 (cooling by liquid nitrogen, heating by peltier). There is no heat exchanger but a bottle with liquide nitrogen with a control system

lab. 3 Das Gerät wird einmal jährlich durch den Gerätehersteller gewartet und justiert. Quartalsweise erfolgt eine Messung der Trägheit/Luftdurchströmung/Abweichung des Drehmomentes/Messung eines Standardviskosen Material bei 70°C und Aufzeichnung in einer Kontrollkarte.

lab. 7 Compliance correction has been determined in a previous step, sample temperature has been measured and corrected for

lab. 10 Compliance specified in 2.9.1 is for lower measuring system geometry (it is put in the DSR cell of "torsional compliance". The entire compliance is 0.024 (0.0016 is for rheometer motor).
We have already published a paper on 4-mm DSR "Analysis of 4-mm DSR tests: calibration, sample preparation, and evaluation of repeatability and reproducibility" and Judita Gražulytė have done tests with 4-mm DSR in her doctoral thesis "Bitumen stress relaxation modulus as an indicator of asphalt pavements resistance to low temperature cracking".
At the end is a summary in english:
http://dspace.vgtu.lt/bitstream/1/3824/1/J.%20Gra%c5%beulyt%c4%97s%20disertacija.pdf

lab. 13 The used 4mm-plate-plate-geometry is sand-blasted.

lab. 16 But not so much experiences (4mm-geometry), about under 20 measurements. We did not insert the compliance correction into the software. The calculation is done manually. Sample 1 field has raw result and field of sample 2 is having compliance corrected value. So far the sample 2 was not run. I will get back to you about the countercooling. We have problems with going below the 0 deg C of the coolant. The room is small and with no great heat exchange of its own, I suppose Julabo is not as effective in cooling it down. So the lowest stable temperature we can go down to with our system is-28

6.2 Sample preparation

laboratory	At which temperature has the gap set zero?	Has a sub-sample of the distributed container been taken for sample preparation?
	[°C]	(YES/NO)
1	25	YES
2	0	NO
3	-15	YES
4	-15	YES
5	25	YES
6	30	NO
7	-10	NO
8	0	NO
9	25	NO
10	0	YES
11	-15	YES
12	-15	-
13	25	NO
14	-15	NO
15	20	NO
16	5	NO
17	30	NO
18	-	YES
19	25	YES
20	25	NO
21	25	YES
22	0	NO

If NO, describe sample preparation procedure

lab. 2 Sample preparation according to AL DSR / ASHTOO - Samples were heated up to 180 °C and poured into silicone moulds, cooled down at room temperature for 2h. Then the first sample 50/70 was tested and aftwards the second one, which was stored at room temperature. The two samples of 25/55-55A, which were prepared at the same time, were stored in a fridge over night at 5-8°C and tested at the next day.

lab. 6 Pouring hot bitumen into moulds from two-component silicone rubber (Lukopren N 1522)

lab. 7 Can was heated and a drop was placed in a mold- see further

lab. 8 Poured on silicon moulds

lab. 9 Bitumen container is warmed up at a low temperature and sample is placed directly on the geometry.

lab. 13 Heat Up Sample to 90°C

lab. 15 No mould used. A small amount of bitumen taken from the container

lab. 16 50/70 container heated 135 C at 1 h and 25/55-55 container heated 140 C at 1 h-> stirr and molds

lab. 17 Sample was heated up to 170°C for 1 hour and poured into silicone moulds.

lab. 22 1. Sample was taken by tweezers from container. (in 25 oC) 2. The portion of sample was weighted and formed into cylinder shape of 4mm diameter. This act was made by fingers (fresh super nitrile gloves was used separately for each sample as a protection from "fat"). Tools used: ruler, steel spatula, calliper.

https://doi.org/10.24355/dbbs.084-202111031047-0

laboratory	If YES, approximate weight of the sub-sample	If YES, heating temperature of the sub-sample	If YES, heating time of the sub-sample	Have the sub samples been used for direct application in the rheometer?
	[g]	[°C]	[min]	(YES/NO)
1	50	160	5	YES
2	-	-	-	NO
3	10	145	60	NO
4	50	165	15	NO
5	70	150	90	YES
6	-	-	-	NO
7	-	-	-	-
8	-	-	-	NO
9	-	-	-	-
10	17	150 for 50/70; 170 for 25/55-55	20	NO
11	50	150	45	NO
12	-	-	-	NO
13	-	-	-	NO
14	-	-	-	-
15	-	-	-	NO
16	-	-	-	-
17	-	-	-	-
18	125	140	60	YES
19	30	150	1	NO
20	-	-	-	-
21	15	135	1	NO
22	-	-	-	NO

laboratory	Were moulds or sheet materials used for specimen preparation?	If YES, specify material of mould or sheet	If YES, specify shape of the specimen	If YES, storage time before demoulding and testing
	(YES/NO)			[min]
1	YES	Silicone	Rectangle	5
2	YES	Silicon	like ASHTOO 315 with 4mm diameter	see 3.2.1
3	YES	Silicone mold as in AL DSR (T-Sweep / BTSV)	diameter 8 mm and 2 mm height	720
4	YES	Silicon	4mm diameter, height 2.5 mm	30-240
5	YES	Malvern, silicone mould	Cylindrical	0
6	YES	two-component silicone rubber (Lukopren N 1522)	Cylindrical	15
7	YES	Silicon molds	2.5 mm height, 4.2 mm diameter	first repeat 5 min, second repeat about 4 hours
8	YES	Silicon	cylinder	60
9	NO	-	-	-
10	YES	Silicone	cylindrical	15
11	YES	Silicone	Circular, with a diameter of 4mm	720
12	YES	Non sticky silicon sheet material	-	-
13	YES	Silicone Rubber	Cylindrical Shape	
14	NO	-	-	-
15	NO	-	-	-
16	YES	Silicone moulds	Cylinder for 8 mm plate	50/70: 2h 41 min and 25/55-55: 19h 10 min
17	-	-	-	-
18	YES	Silicone moulds	cylinder	12H
19	YES	Silicone	25mm	20
20	-	-	-	-
21	YES	Silicon	AAHSTO mold 8mm	12 h
22	NO	-	-	-

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laboratory	If YES, specimen placed in a refrigerator prior to demoulding?	If YES, duration of conditioning in refrigerator	Sample loading temperature 50/70	Sample loading temperature 25/55-55	How were the sample loading temperatures chosen? (e.g. based on experience, based on softening point)
	(YES/NO)	[min]	[°C]	[°C]	
1	YES	2	60	60	Based on experience
2	NO	-	60	70	We used the same bitumen in FE 07.0293
3	NO	-	50	50	Experience
4	YES	10	80	70	based on softening point
5	NO	-	10	10	Sample was poured directly into the mould at 90°C. Then cooled down to 10°C. Temeperature is based on literature survey.
6	NO	-	30	30	Based on experience
7	NO	-	50	50	experience, and visually after the test when taking out the sample
8	NO	-	48	72	based on softeninf point
9	-	-	25	30	based on experience
10	NO	-	70	70	based on experience
11	YES	< 5	60 then 40	60 then 40	Thanks the ball-ring softening point of bitumen (60°C) and for final snatching (40°C)
12	YES	-	45	50	Based on softing point
13	NO	-	90	90	based on experience
14	-	-	45	55	Softening Point
15	-	-	40	40	Based on experience
16	No	-	45	50	experience
17	-	-	30	30	Based on the cited article
18	YES	15	45	50	softening point
19	NO	-	80	90	experience
20	-	-	62	67	softening point
21	NO	-	50	60	softening point
22	-	-	60	70	both- experience + softening point

laboratory	Equilibrium time of loading temperature before sample loading	Sample loading gap (desired gap + closure gap for the bulge)	Equilibrium time of loading temperature after sample loading
	[min]	[mm]	[min]
1	1	3.5	1
2	-	2	ca. 5
3	5	1.8	10
4	10	1.525	10
5	5	0	10
6	5	2.1	10
7	5	1.80 mm after trimming 1.75mm	5
8	5	4.5	0
9	10	1.1	10
10	5	1.870	2
11	15	1.825	< 5
12	5	2.025	5
13	1	3	2
14	10	1.87	5
15	5	10	5
16	10	1.775	5
17	-	2.05	10
18	5	1.775	30
19	1	-	1
20	1	1.87	5
21	0	2.1	1
22	1	2.050 mm stick gap	0.5

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laboratory	Has the specimen been trimmed after loading in the DSR?	If YES, approximate temperature of trimming tool	If YES, specify material of trimming tool (e.g. plastic, aluminum, stainless steel)	If YES, specify shape of trimming tool
	(YES/NO)	[°C]		
1	YES	10	stainless steel	Chisel
2	YES	100	stainless steel	spatula
3	YES	90	stainless steel	Spatel mit Radius
4	YES	<90	stainless steel	90° bended spetula
5	NO	-	-	-
6	YES	100	Stainless steel	Bent spatula
7	YES (at 15°C)	the spatula is hold against a hot stage at 180°C, so my guess is at 160°C	stainless steel	standard spatula (not curved!) area is 2cm on 0.5 cm
8	YES	160	stainless steel	creased chemical spatula
9	YES	90	stainless steel	Scalpel
10	YES	95	aluminum	spatula
11	YES	40	stainless steel	With curved and linear axis
12	YES	-	stainless steel	L shape
13	YES	90	stainless steel	rectangular
14	YES	100	stainless steel	spatula
15	YES	Unknwon	Stainless steel	L
16	YES	??75	stainless steel	dogbone with flat end
17	YES	25	stainless steel	spatula
18	YES	150	stainless steel	spatula
19	YES	140	stainless steel	section of a ruler
20	NO	-	-	-
21	YES	120	stainless steel	bended spatula
22	NO	-	-	-

laboratory	Has the specimen the exact weight, so trimming is not necessary?	If YES, weigth of the specimen	Final gap after sample preparation
	(YES/NO)	[mg]	[mm]
1	NO	-	3
2	NO	-	1.75
3	NO	-	1.75
4	NO	-	1.5
5	YES	-	3
6	NO	-	2
7	NO	-	1.75
8	NO	-	2.2
9	NO	-	1
10	NO	-	1.75
11	NO	-	1.75
12	NO	-	2
13	NO	-	ca. 2.7
14	NO	-	1.75
15	NO	-	3
16	NO	-	1.75
17	NO	-	2
18	NO	-	1.75
19	NO	-	3.1 ± 1.0
20	YES	0.025	1.75
21	NO	-	2
22	YES	0.0265	1.9

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If available: references for sample preparations method	
lab. 2	FE 07.0293
lab. 4	AASHTO T315
lab. 5	Schrader et al. 2019: On low temperature binder testing using 4 mm geometry, EATA 2019
lab. 7	J. Gražulytė, H. Soenen, J. Blom, A. Vaitkus, J. Židanavičiūtė & A. Margaritis (2019): Analysis of 4-mm DSR tests: calibration, sample preparation, and evaluation of repeatability and reproducibility, Road Materials and Pavement Design, DOI: 10.1080/14680629.2019.1634636
lab. 8	AASHTO project: determining the low temperature rheological properties of asphalt binder using a dynamic shear rheometer
lab. 10	Samples preparation method is published in "Analysis of 4-mm DSR tests: calibration, sample preparation, and evaluation of repeatability and reproducibility": https://www.tandfonline.com/doi/pdf/10.1080/14680629.2019.1634636?needAccess=true
lab. 14	Western Research Institute sample preparation protocol
lab. 16	We follow what was in AASHTO draft in 2015. Trimming with hot spatula.
lab. 17	ČSN EN 14 770, LU, Xiaohu; UHLBACK, Petri; SOENEN, Hilde. Investigation of bitumen low temperature properties using a dynamic shear rheometer with 4 mm parallel plates. International Journal of Pavement Research and Technology, 2017, 10.1: 15-22.
lab. 19	Wang, D., Cannone Falchetto, A., Alisov, A., Schrader, J., Riccardi, C. & Wistuba, M. P. 2019. An Alternative Experimental Method for Measuring the Low Temperature Rheological Properties of Asphalt Binder Using 4 mm Parallel Plates on Dynamic Shear Rheometer. Transportation Research Record: Journal of the Transportation Research Board, Vol. 2673, Issue 3, 427–438. DOI: 10.1177/0361198119834912. Schrader, J., Wistuba, M. P., Remmler, T. & Wang, D. 2019. On Low Temperature Binder Testing using DSR 4mm Geometry, under review. Materials and Structures, Springer.
lab. 20	Farrar, M., Sui, C., Salmans, S., & Qin, Q. (2015). Determining the low-temperature rheological properties of asphalt binder using a dynamic shear rheometer (DSR). Fundamental Properties of Asphalts and Modified Asphalts III Product: FP, 8.

Comments	
lab. 5	The subsample was heated several times in order to evaluate LVE. The subsample was also used for the evaluation of the needed conditioning time. The subsample could aged a little bit over time. For the subsequent measurements a new subsample was used.
lab. 7	temp interval from +10°C to-30°C
lab. 8	loading at R&B +2°C and just after loading, cooling at 15°C. equilibrium time at 15°C: 5min. First trimming at 4.5mm. 2nd triming at 2.4mm.
lab. 13	Final Gap achieved under normal force control
lab. 14	The material was transferred to the lower fixed plate by means of a hot spatula- the upper plate was lowered to 1.87 mm- the sample was trimmed- the temperature was lowered to 30°C and the gap was closed to 1.75 mm
lab. 15	The samples were trimmed at 0°C (10 min of equilibrium time). It was very difficult to get a proper specimen geometry. We tried different methods but we are still not satisfied with the results. Further tests are needed.
lab. 16	we used 8mm moulds and samples were done smaller after moulding with hot spatula.
lab. 18	* Use ever the same temperature of the analisys
lab. 19	Sample height is trimmed instead of sample radius at 10°C
lab. 22	Better results are obtained by using 1.9mm gap rather than 2.0 mm Better results are obtained by using sample volume or weight rather than its triming

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6.3 Sample conditioning and test parameters

laboratory	Was an increasing trend used for subsequent test temperatures?	Was the normal force kept zero <u>during temperature conditioning</u> for each of the test temperatures?	Was the gap kept constant <u>during testing</u> for each of the temperatures?
	(YES/NO)	(YES/NO)	(YES/NO)
1	YES	YES	YES
2	NO	YES	NO
3	NO	YES	YES
4	NO	NO	NO
5	NO	YES	NO
6	NO	NO	YES
7	NO	YES	YES
8	NO	YES	NO
9	NO	NO	YES
10	NO	YES	NO
11	NO	YES	YES
12	NO	YES	YES
13	YES	YES	YES
14	NO	YES	YES
15	NO	YES	NO
16	NO	YES	NO
17	YES	NO	YES
18	NO	NO	YES
19	YES	YES	YES
20	NO	YES	YES
21	YES	YES	YES
22	NO	NO	YES

laboratory	Duration of equilibrium time at test temperature-30°C before measure	Duration of equilibrium time at test temperature-20°C before measure	Duration of equilibrium time at test temperature-10°C before measure	Duration of equilibrium time at test temperature 0°C before measure
	[min]	[min]	[min]	[min]
1	1	1	1	1
2	-	30	30	30
3	10	10	10	10
4	10	10	10	10
5	60	60	60	60
6	20	20	20	20
7	20	20	20	20
8	2	2	2	2
9	10	10	10	10
10	20	20	20	20
11	15	15	15	15
12	5	5	5	5
13	45	30	20	10
14	5	5	5	10
15	-	See comment	10	10
16	15	15	15	15
17	20	10	10	10
18	30	30	30	30
19	45	30	20	10
20	40	40	40	40
21	45	30	20	10
22	10	10	10	10

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laboratory	Have the equilibrium times been selected based on Annex B of EN 14770?	Has the thermal expansion of the geometry been checked during the last 8 weeks	Has gas/air been used to prevent ice formation at the interface between the geometry and the temperature hood?	If YES, specify the type of gas/air and the volume flow
	(YES/NO)	(YES/NO)	(YES/NO)	
1	-	YES	YES	LIQUID NITROGEN
2	NO	YES	NO	-
3	NO	YES	Yes, Peltier	standard-Peltier
4	NO	YES	YES	dried O2 220Ln/h
5	NO	NO	NO	-
6	NO	NO	NO	-
7	No (determined with thermocouples)	NO	YES	dry air/ 200l/h
8	NO	NO	YES	comprimed air at 200l/h
9	NO	YES	NO	-
10	YES	NO	YES	air, 200 l_N/h
11	YES	NO	YES	Compressed air- 2 bar
12	-	-	-	-
13	NO	YES	YES	Dried Air
14	NO	YES	NO	-
15	NO	NO	NO	-
16	No or I don't know	NO	-	-
17	NO	NO	NO	-
18	YES	YES	YES	air 200 L_n/h
19	YES	YES	YES	nitrogen 99.999%, 2 L/min
20	YES	YES	YES	nitrogen 99.999%, 2 L/min
21	YES	YES	NO	-
22	NO	YES	NO	-

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laboratory	Has the linear viscoelatic (LVE) range been determined for the materials? (YES/NO)	If YES, specify tested temperatures, tested frequencies and tested stress/strain amplitude	If NO, has the strain/stress been selected based on previous experience with the materials? (YES/NO)	Is strain or stress controlled mode used for the T-f-Sweep?
1	YES	-	-	Stress
2	NO	-	YES	Strain
3	YES	strain at-30°C and 10 Hz	YES	Strain
4	YES	0°C,-30°C, 1.59Hz, 0.01-10%	-	Strain
5	YES	Temperatures: 0°C and 30°C, tested frequencies: 0.1Hz and 10 Hz, LVER Stress mode 1 kPa to 1000 kPa	-	Stress
6	YES	0°C and 30°C, 0.1Hz and 10 Hz, 0.1 kPa to 1000 kPa	-	Stress
7	NO	-	YES	Strain
8	NO	-	YES	Strain
9	YES	0,-10,-20,-30°C; 0.1, 1.0, 1.59, 10 Hz; 0.01- 0.5 % strain	-	Strain
10	YES	at 0°C with 0.1Hz; at –30°C with 10 Hz; in both cases strain from 0.0001 to 0.1	-	Strain
11	YES	0°C,-10°C,-30°C ; 1.59Hz ; from 1E-5 % to 1 %	-	-
12	NO	-	NO	-
13	NO	-	YES	Stress
14	NO	-	YES	Strain
15	NO	-	YES	Strain
16	No	-	YES	-
17	YES	0°C, 0.1- 10 Hz, strain 10^{-6}- 10^{-4} %, stress 0- 10 Pa	NO	Stress
18	YES	-	-	-
19	NO	-	YES	Stress
20	YES	-30C with 10Hz and 0C with 0.1Hz	-	Stress
21	NO	-	YES	Stress
22	NO	-	YES	Strain

laboratory	Strain or stress applied							
	-30°C (50/70)	-20°C (50/70)	-10°C (50/70)	0°C (50/70)	-30°C (25/55-55)	-20°C (25/55-55)	-10°C (25/55-55)	0°C (25/55-55)
	% / kPa	% / kPa	% / kPa	% / kPa	% / kPa	% / kPa	% / kPa	% / kPa
1	10	10	10	10	10	10	10	10
2	-	0.2 (1.0 Hz) 0.1 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)	0.2 (1.0 Hz) 0.2 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)	0.2 (1.0 Hz) 0.2 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)	-	0.2 (1.0 Hz) 0.1 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)	0.2 (1.0 Hz) 0.2 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)	0.2 (1.0 Hz) 0.2 (1Hz) 0.1 (1.59Hz) 0.05 (10Hz)
3	0.005	0.005	0.005	0.0083	0.005	0.005	0.005	0.0084
4	0.1	0.1	0.1	0.1	0.05	0.05	0.05	0.05
5	10 and 100	10 and 100	10 and 100	10 and 100	10 and 100	10 and 100	10 and 100	10 and 100
6	50	50	50	50	50	50	50	50
7	0.02	0.02	0.02	0.02	0.02	0.02	0.02	0.02
8	0.01	0.01	0.01	0.01	0.01	0.01	0.01	0.01
9	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
10	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
11	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
12	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
13	300	100	10	5	300	100	10	5
14	0.05	0.05	0.05	0.05	0.05	0.05	0.05	0.05
15	-	-	0.30	0.40	-	-	0.20	0.30
16	0.1	0.1	0.1	0.1	0.1	0.1	0.1	0.1
17	1	1	1	1	1	1	1	1
18	-	0.001	0.01	0.5	-	0.001	0.1	0.1
19	100	50	25	10	100	50	25	10
20	10	10	10	10	10	10	10	10
21	100	50	25	10	100	50	25	10
22	0.01026	0.01026	0.01026	0.01026	0.01026	0.01026	0.01026	0.01026

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laboratory	Measurement duration for each data point					Date of testing the 50/70	Date of testing the 25/55-55
	Number of individual measurement values for each temperature-frequency combination	0.10 Hz	1.00 Hz	1.59Hz	10.00Hz		
	[-]	[s]	[s]	[s]	[s]		
1	5	100	10	6.3	1	07.10.2019	10.10.2019
2	0.1Hz 5Measurements; Rest 10	45	15	15	10	30.09.2019	01.10.2019
3	n+10+1	auto				08.10.2019	09.10.2019
4	1	ca.74	ca.13	ca.10	ca.8	10.09.2019	12.09.2019
5	4	117	47	27	11	11. and 12.11.2019	13. to 15.11 2019
6	10	10	10	10	10	13. to 15.11.2019	18. to 20.11.2019
7	automatic-software decides	77	26	20	18	12.11.2009	13.11.2019
8	1	10	10	10	10	07.11.2019	12.11.2019
9	5	240	240	240	240	KW45	KW46
10	5	108	16	14	6	08.11.2019	11.11.2019
11	15	10	1	0.6	0.1	13. and 14.11.2019	13. and 15.11.2019
12	-	-	-	-	-	15.11.2019	18.11.2019
13	1	300	30	20	20	-	-
14	1	-	-	-	-	09.10.2019	11.10.2019
15	By the equipment (Automatic)					22.11.2019	22.11.2019
16	1	set by device				13.11.2019	15.11.2019
17	20	5	5	5	5	14.11.2019	18.11.2019
18	26	*	*	*	*	7 to 11.10.2019	22 to 29.10.2019
19	1	180	30	20	20	05.- 06.09.2019	05.- 06.09.2019
20	1	120	10	9	3	2019.10.20	2019.10.20
21	1	180	30	20	20	14.11.2019	14.11.2019
22	6	15.32	5.7	5.7	6.1	9.- 10.01.2020	10.01.2020

Comments

lab. 1 Duration of the whole temperature-frequency combination equals 16 minutes.

lab. 3 Die Deformation wurde Automatisch am Gerät über eine einprogrammierte Abhängigkeit zwischen LVE-Grenze und der gemessenen Spannung eingestellt. Die Abhängigkeit wurde im FE NaHiTas des BMBV abgeleitet und ist dort beschrieben.

lab. 5 Based on LVE measurements two stress levels were applied during the measurements. A sequence was prepared so to measure 10 kPa firstly then 100 kPa and once again 10 kPa and 100 kPa in order to check whether the sample was not damaged during testing. If the values are close enough we know that the sample is still intact. In this way we were able to track damaged samples. LVE was measured only on 50/70.

lab. 7 measuring duration is the sum of adjusting and averaring time (this was set on automatic)

lab. 10 Test temperatures were from highest (0°C) to lowest (-30°C).

lab. 11 First tests with the 4 mm plate geometry by our laboratory. The geometry wasn't used before these tests

lab. 13 I did not find the time for amplitude sweeps, just checked harmonic distortion values to be within acceptable limits.

lab. 15 We are presenting only the results at 0°C and-10°C. At lower temperatures (-20°C) the specimen debonded. We also tried to perform some tests are lower strain levels but we were not happy with the results.

lab. 16 we used-28 C not-30 C, and also 10 C was measured 10->-28. Peltier is connected to pressurised air and it is kept at around 180 L/h. We have done the LVE ranges for other bitumens with and without modification previously to choose the strain.

lab. 17 In a first approach, there was an effort to measure completely according to the article LU, Xiaohu; UHLBACK, Petri; SOENEN, Hilde. Investigation of bitumen low temperature properties using a dynamic shear rheometer with 4 mm parallel plates. International Journal of Pavement Research and Technology, 2017, 10.1: 15-22.1). However, some parameters were necessary to be omitted/modified: 1) Normal force control (0 N during temperature equlibrion) was not able to be used, however there were only normal forces up to 0,05 N. 2) Temperature equilibrium was necessary to be prolonged to 20 minutes in case of measurements at-30°C. 3) The stress level was 1 MPa for the sample 50/70 but it had to be lowered to 1 kPa for the sample 25/55-55 (instrument limits were exceeded). 4) Freezing of the samples before the measurement was not carried out. 5) Linear viscoelastic range was determined, however according to the cited article, fixed stress level was used (1 MPa) and consequently it was lowered for the sample 25/55-55 (1 kPa).

lab. 18 * We use the function "set by device" so We have a variable number of representative point.

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6.4 Rheological parameters

laboratory	Have additional test results beyond the below stated been discarded?	If YES, why?	Have the results been compared to 8mm-geometry measurements?	If YES, what was the maximum percentual difference of shear modulus
	(YES/NO)		(YES/NO)	
1	NO	-	NO	
2	YES	Because our T-Sweep is from 30°C to-20°C	NO	
3	NO	-	NO	
4	YES	Different operators, thus different values	NO	
5	YES	Samples were damaged during testing. We had severe problems with freezing. Therefore, the results below contain only the results which we think are reliable (to a certain point).	NO	
6	YES	Large differences between measurements, inappropriate shear stress	NO	
7	YES	data at 10°C-not asked for	NO	
8	NO	-	NO	
9	NO	-	NO	
10	NO	-	NO	
11	YES	Testing different gap for fixed the final gap for these tests.	NO	
12	NO	-	NO	
13	NO	-	NO	
14	NO	-	NO	
15	YES	We are gaining experience with this testing method. There are still a lot of uncertainties associated with the specimen geometry. We performed several tests (different specimen preparation techniques, different strain levels) The phase angle (who is independent of the geometry) was very similar between the tests. However, there was a lot of variation in the dynamic shear modulus	NO	
16	NO	-	Not yet	
17	YES	Because the first measurement of the sample 25-55-55 at the stress level 1 Mpa lead to error in measurement causing the occurrence of non-representative results. Therefore, only results at the stress level 1 kPa are involved.	NO	
18	YES	no repeatibility	NO	
19	YES	Ice formation at-20°C	YES	17%
20	-	-	NO	
21	NO	-	NO	
22	YES	Test was prepared for 6 individual measurements (slightly scatter of results was observed- module value) + extra tests which allowed create repetitive procedure for used device for both spindles (4 and 8 mm)	YES	5 %

Comments

Lab. 1 We have a technical posibilities to perform test in-30 but bitumen specimens lost bond with steal plates when they coold down to-30. Results were unrealistic thus we did not includ them. We suppose that less strain should be applied, however to confirm it we would need more time.

Lab. 5 Measurements will be reapeted as soon as the right equipement is placed into a working condition. There will be either nitrogen or dry air.

Lab. 10 samples were tested from the highest frequency to the lowest one.

Lab. 11 During the second sample for 25/55-55, we had a problem with our viscotherm VT 2, that stop current test. Because of the condensation on the pipe, the bath lost liquid, so we must to add a bit of mixing liquid (50 glycol/50 water) and restart the test with the same sample.
The rheometer was stop current test when peltier plan was dropped in temperature. In the beginning of the test, it was dropping from 60°C to 0°C, and after the incident we restart from 21°C to 0°C, because 21°C was the temperature of the peltier plan when the test was stopped.

Lab. 13 Rather than cooling with maximum cooling rate from loading temperature to first test temperature, I think we should define a specific cooling rate . I did not find the time to make a comparison of reproducability between rapid, undefined cooling and defined cooling with small cooling rate. It looks like there is a structural impact present.

Lab. 15 We have some concerns about the magnitude of the shear modulus determined with the PP4. It seems too high.

Lab. 16 we used-28 C not-30 C, and also 10 C was measured 10->-28

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6.5 Statistical analysis of rheological parameters @1.59Hz

6.5.1 Complex Shear Modulus G*

Characteristic: complex shear modulus G*
Material: 50/70
Frequency: 1.59Hz
Temperature: -30°C

laboratory	value 1 [kPa]	value 2 [kPa]	mean value [kPa]	deviation [%]	lab deviation [%]	z score
11	335 268	312 420	323 844	3.53	-53.30	-2.27
17	342 707	475 787	409 247	16.26	-40.99	-1.75
4	552 430	543 180	547 805	0.84	-21.01	-0.90
12	568 480	543 100	555 790	2.28	-19.86	-0.85
3	596 744	562 533	579 639	2.95	-16.42	-0.70
6	580 400	616 310	598 355	3.00	-13.72	-0.58
13	627 448	713 064	670 256	6.39	-3.35	-0.14
9	665 844	712 635	689 240	3.39	-0.61	-0.03
16	758 167	713 094	735 630	3.06	6.08	0.26
7	757 300	798 200	777 750	2.63	12.15	0.52
14	814 120	806 720	810 420	0.46	16.86	0.72
20	840 371	799 973	820 172	2.46	18.27	0.78
19	849 014	795 945	822 480	3.23	18.60	0.79
10	852 320	846 760	849 540	0.33	22.50	0.96
21	890 794	810 000	850 397	4.75	22.62	0.96
22	934 338	791 248	862 793	8.29	24.41	1.04
8	852 710	919 400	886 055	3.76	27.77	1.18
5	2 115 219	-	2 115 219	0.00	205.01	8.74
		general mean:	693 495			

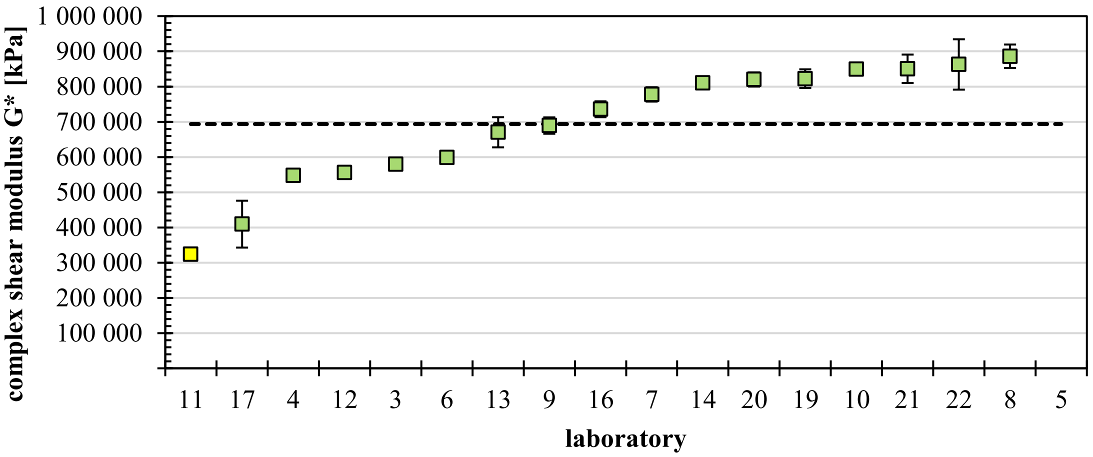

Figure 1 Complex shear modulus G* of plain binder 50/70 at 1.59Hz and -30°C

Lab 1, 2, 15 and 18 were not able to test at test temperature-30°C.
Lab 5 only provided one test result.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: **complex shear modulus G***
Material: **50/70**
Frequency: **1.59Hz**
Temperature: **-20°C**

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[kPa]	[kPa]	[kPa]	[%]	[%]	
11	245 382	233 374	239 378	2.51	-51.51	-2.25
17	204 661	362 914	283 788	27.88	-42.51	-1.86
6	387 110	411 060	399 085	3.00	-19.15	-0.84
1	399 678	399 497	399 588	0.02	-19.05	-0.83
12	427 150	406 500	416 825	2.48	-15.56	-0.68
2	435 467	408 196	421 832	3.23	-14.55	-0.64
3	457 588	426 869	442 229	3.47	-10.42	-0.45
9	440 153	447 137	443 645	0.79	-10.13	-0.44
4	453 080	450 230	451 655	0.32	-8.51	-0.37
18	467 430	502 412	484 921	3.61	-1.77	-0.08
13	470 725	504 629	487 677	3.48	-1.21	-0.05
7	525 700	554 500	540 100	2.67	9.41	0.41
16	578 037	541 611	559 824	3.25	13.41	0.59
22	641 683	550 418	596 050	7.66	20.75	0.91
14	599 250	596 720	597 985	0.21	21.14	0.92
21	638 832	570 000	604 416	5.69	22.44	0.98
10	610 260	600 700	605 480	0.79	22.66	0.99
20	632 430	613 705	623 068	1.50	26.22	1.14
8	605 480	657 430	631 455	4.11	27.92	1.22
19	669 080	618 604	643 842	3.92	30.43	1.33
5	1 325 483	-	1 325 483	0.00	168.51	7.36
		general mean:	493 642			

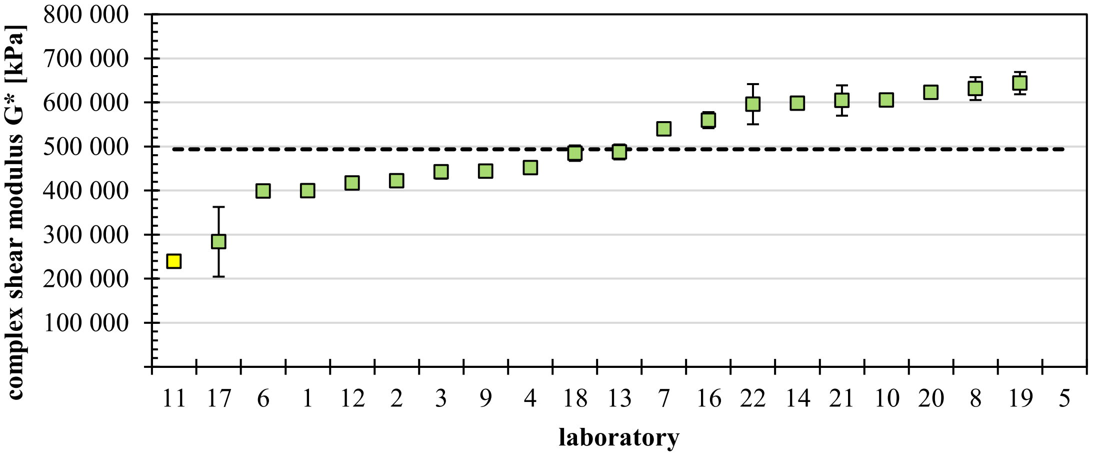

Figure 2: complex shear modulus G* of plain binder 50/70 at 1.59Hz and -20°C

Lab 15 was not able to test at test temperature-20°C.
Lab 5 only provided one test result.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: complex shear modulus G*
Material: 50/70
Frequency: 1.59Hz
Temperature: -10°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[kPa]	[kPa]	[kPa]	[%]	[%]	
11	116 105	118 654	117 380	1.09	-52.11	-2.18
17	144 687	146 057	145 372	0.47	-40.69	-1.70
1	156 641	160 536	158 589	1.23	-35.29	-1.48
6	198 490	210 760	204 625	3.00	-16.51	-0.69
2	209 785	202 064	205 925	1.87	-15.98	-0.67
12	211 410	202 220	206 815	2.22	-15.62	-0.65
13	210 162	215 979	213 071	1.37	-13.06	-0.55
5	228 935	-	228 935	0.00	-6.59	-0.28
3	250 132	232 521	241 327	3.65	-1.54	-0.06
9	242 947	246 060	244 504	0.64	-0.24	-0.01
7	239 400	253 900	246 650	2.94	0.64	0.03
18	273 968	234 634	254 301	7.73	3.76	0.16
4	263 390	266 670	265 030	0.62	8.14	0.34
22	279 428	251 213	265 320	5.32	8.25	0.34
10	272 620	269 930	271 275	0.50	10.68	0.45
21	287 220	258 000	272 610	5.36	11.23	0.47
16	287 771	269 320	278 546	3.31	13.65	0.57
8	271 790	296 600	284 195	4.36	15.96	0.67
14	290 220	285 970	288 095	0.74	17.55	0.73
20	300 445	278 936	289 691	3.71	18.20	0.76
19	353 795	277 749	315 772	12.04	28.84	1.21
15	418 420	369 500	393 960	6.21	60.74	2.54
		general mean:	245 090			

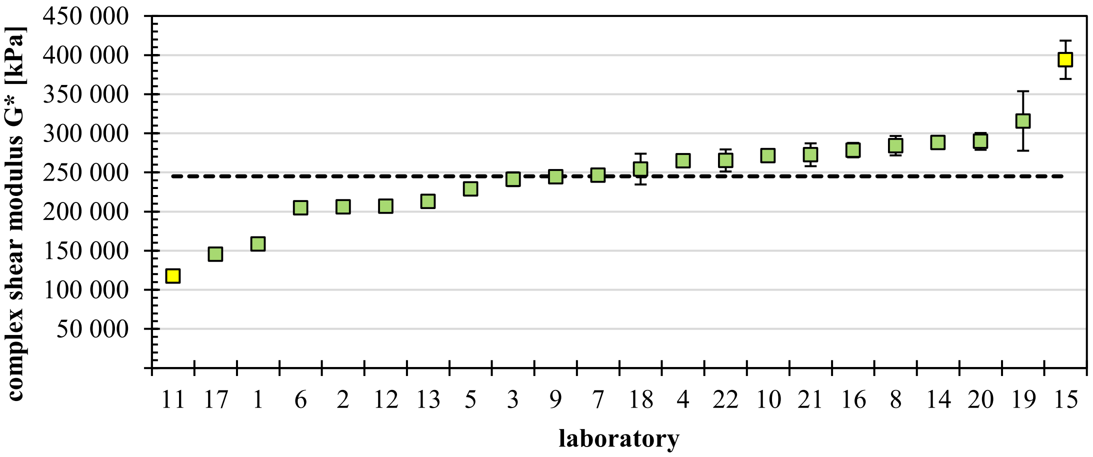

Figure 3: complex shear modulus G* of plain binder 50/70 at 1.59Hz and-10°C

Lab 5 only provided one test result.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: complex shear modulus G*
Material: 50/70
Frequency: 1.59Hz
Temperature: 0°C

laboratory	value 1 [kPa]	value 2 [kPa]	mean value [kPa]	deviation [%]	lab deviation [%]	z score
11	38 654	43 166	40 910	5.51	-44.57	-1.67
1	43 346	43 537	43 441	0.22	-41.14	-1.54
13	48 851	48 893	48 872	0.04	-33.78	-1.27
6	48 340	51 320	49 830	2.99	-32.48	-1.22
17	52 994	52 525	52 759	0.44	-28.51	-1.07
5	65 385	51 802	58 594	11.59	-20.60	-0.77
18	67 128	64 840	65 984	1.73	-10.59	-0.40
2	67 118	66 134	66 626	0.74	-9.72	-0.36
12	67 970	65 770	66 870	1.64	-9.39	-0.35
21	72 677	67 600	70 138	3.62	-4.96	-0.19
9	70 052	78 916	74 484	5.95	0.93	0.03
7	75 260	80 280	77 770	3.23	5.38	0.20
3	81 829	77 608	79 718	2.65	8.02	0.30
22	84 562	79 280	81 921	3.22	11.01	0.41
10	83 210	83 755	83 483	0.33	13.12	0.49
20	87 485	81 211	84 348	3.72	14.29	0.54
8	83 022	90 702	86 862	4.42	17.70	0.66
16	93 283	83 231	88 257	5.70	19.59	0.74
19	93 561	83 307	88 434	5.80	19.83	0.74
14	95 233	92 398	93 816	1.51	27.12	1.02
4	93 811	95 343	94 577	0.81	28.15	1.06
15	134 550	117 220	125 885	6.88	70.58	2.65
		general mean:	73 799			

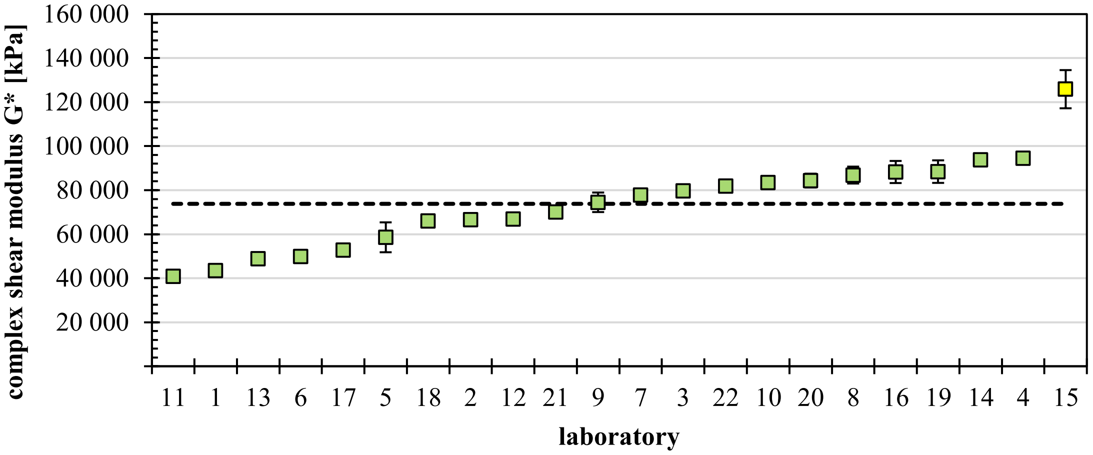

Figure 4: complex shear modulus G* of plain binder 50/70 at 1.59Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59Hz**
Temperature: **-30°C**

laboratory	value 1 [kPa]	value 2 [kPa]	mean value [kPa]	deviation [%]	lab deviation [%]	z score
17	453 815	410 774	432 294	4.98	-37.76	-1.85
4	449 160	431 300	440 230	2.03	-36.61	-1.79
3	461 736	588 585	525 161	12.08	-24.39	-1.19
12	542 500	564 230	553 365	1.96	-20.32	-0.99
18	604 335	589 563	596 949	1.24	-14.05	-0.69
6	608 360	647 320	627 840	3.10	-9.60	-0.47
9	632 790	647 486	640 138	1.15	-7.83	-0.38
14	632 110	653 090	642 600	1.63	-7.48	-0.37
21	697 000	669 000	683 000	2.05	-1.66	-0.08
16	714 937	698 211	706 574	1.18	1.74	0.08
20	731 480	704 502	717 991	1.88	3.38	0.17
13	687 473	774 600	731 037	5.96	5.26	0.26
7	750 300	766 800	758 550	1.09	9.22	0.45
19	776 197	799 807	788 002	1.50	13.46	0.66
10	825 470	819 460	822 465	0.37	18.42	0.90
8	835 220	820 360	827 790	0.90	19.19	0.94
11	849 192	845 355	847 274	0.23	21.99	1.08
5	926 379	-	926 379	0.00	33.38	1.63
22	862 658	993 885	928 271	7.07	33.66	1.65
		general mean:	694 522			

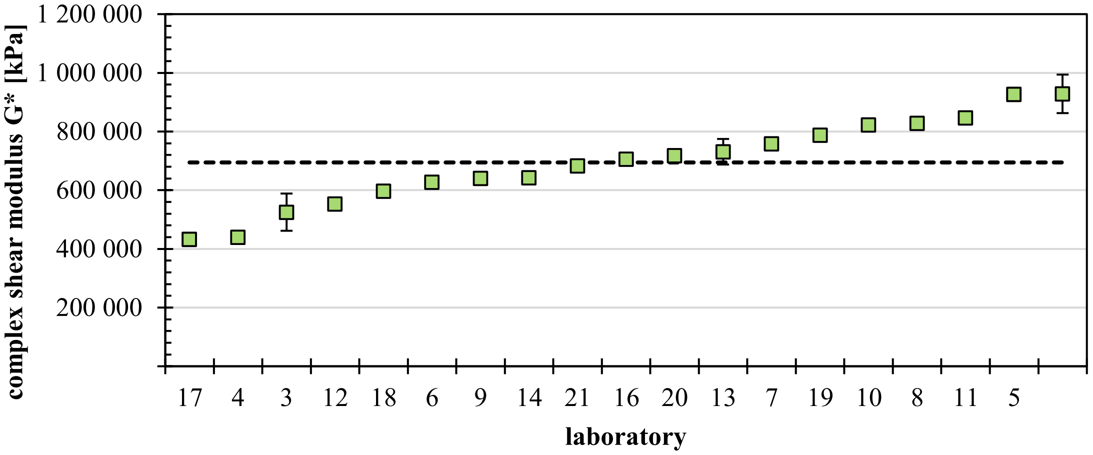

Figure 5: complex shear modulus G* of polymer modified binder 25/55-55 at 1.59Hz and-30°C

Lab 1, 2 and 15 were not able to test at test temperature-30°C.

Lab 5 only provided one test result and is not included in general mean.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: **complex shear modulus G***
Material: **25/55-55**
Frequency: **1.59Hz**
Temperature: **-20°C**

laboratory	value 1 [kPa]	value 2 [kPa]	mean value [kPa]	deviation [%]	lab deviation [%]	z score
17	341 230	293 632	317 431	7.50	-36.06	-1.91
4	361 360	344 110	352 735	2.45	-28.94	-1.54
2	375 464	393 457	384 461	2.34	-22.55	-1.20
1	374 193	449 654	411 924	9.16	-17.02	-0.90
3	366 991	465 955	416 473	11.88	-16.11	-0.85
12	412 200	429 800	421 000	2.09	-15.19	-0.81
6	415 350	441 960	428 655	3.10	-13.65	-0.72
9	419 042	459 212	439 127	4.57	-11.54	-0.61
18	465 403	458 722	462 063	0.72	-6.92	-0.37
14	479 560	486 330	482 945	0.70	-2.71	-0.14
21	506 000	485 000	495 500	2.12	-0.19	-0.01
13	523 354	511 500	517 427	1.15	4.23	0.22
7	537 800	550 700	544 250	1.19	9.63	0.51
20	575 313	525 561	550 437	4.52	10.88	0.58
16	562 829	547 878	555 354	1.35	11.87	0.63
5	560 558	-	560 558	0.00	12.92	0.69
19	575 747	575 320	575 534	0.04	15.94	0.85
10	604 980	602 880	603 930	0.17	21.66	1.15
8	609 890	604 440	607 165	0.45	22.31	1.18
11	637 874	639 499	638 687	0.13	28.66	1.52
22	613 368	705 043	659 205	6.95	32.79	1.74
		general mean:	496 422			

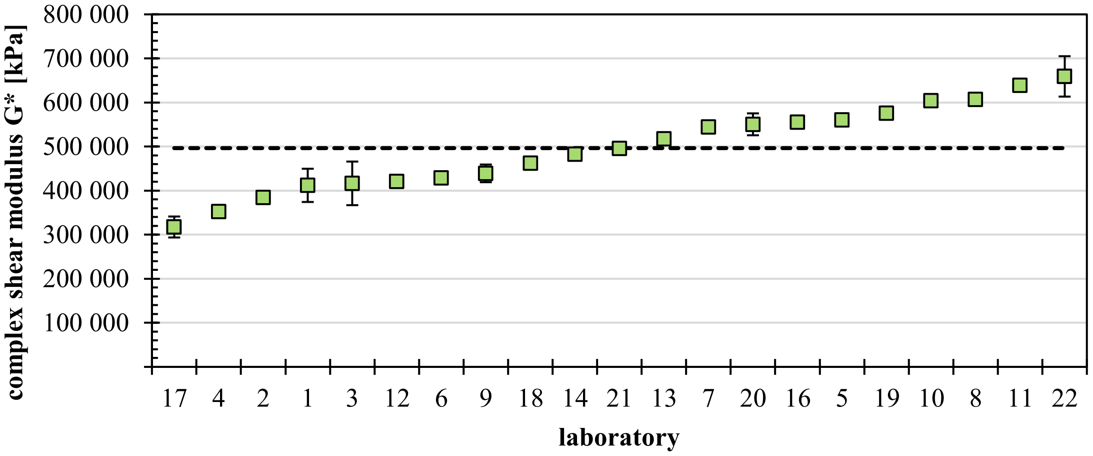

Figure 6: complex shear modulus G* of polymer modified binder 25/55-55 at 1.59Hz and-20°C

Lab 15 was not able to test at test temperature-20°C.

Lab 5 only provided one test result and was included in general mean.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: complex shear modulus G*

Material: 25/55-55

Frequency: 1.59Hz

Temperature: -10°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[kPa]	[kPa]	[kPa]	[%]	[%]	
17	161 522	142 800	152 161	6.15	-39.44	-1.71
2	201 144	207 753	204 449	1.62	-18.63	-0.81
1	185 076	228 814	206 945	10.57	-17.64	-0.76
4	225 980	215 780	220 880	2.31	-12.09	-0.52
12	217 540	226 130	221 835	1.94	-11.71	-0.51
6	229 210	243 970	236 590	3.12	-5.84	-0.25
13	246 682	227 400	237 041	4.07	-5.66	-0.25
3	213 649	268 537	241 093	11.38	-4.05	-0.18
18	243 679	241 564	242 622	0.44	-3.44	-0.15
5	251 955	243 247	247 601	1.76	-1.46	-0.06
21	253 000	243 000	248 000	2.02	-1.30	-0.06
20	249 610	246 412	248 011	0.64	-1.29	-0.06
14	248 540	247 590	248 065	0.19	-1.27	-0.06
9	255 057	250 901	252 979	0.82	0.68	0.03
19	268 301	249 769	259 035	3.58	3.09	0.13
7	266 300	272 300	269 300	1.11	7.18	0.31
10	296 370	294 590	295 480	0.30	17.60	0.76
8	299 100	299 660	299 380	0.09	19.15	0.83
16	306 225	295 478	300 851	1.79	19.74	0.85
22	292 603	337 623	315 113	7.14	25.41	1.10
11	326 897	331 272	329 085	0.66	30.97	1.34
15	461 160	443 160	452 160	1.99	79.96	3.46
		general mean:	251 263			

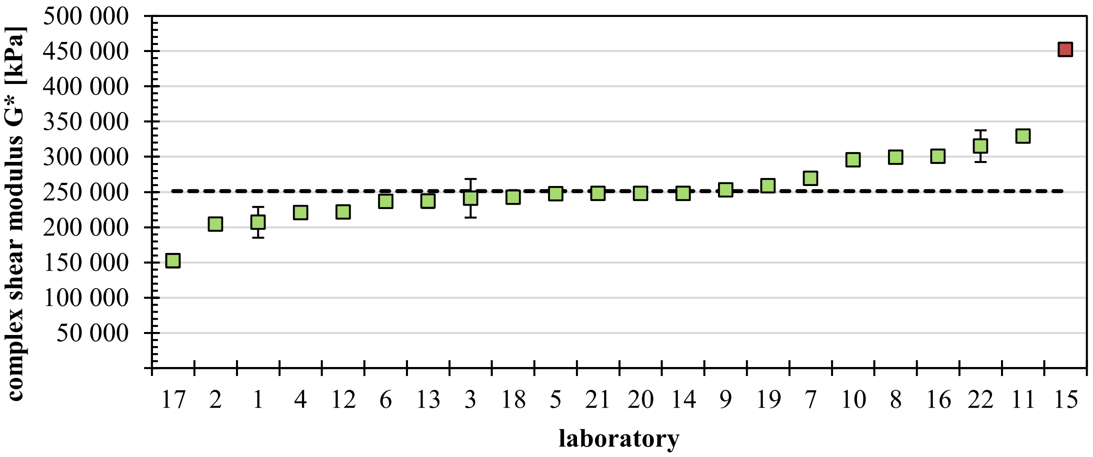

Figure 7: complex shear modulus G* of polymer modified binder 25/55-55 at 1.59Hz and-10°C

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: complex shear modulus G*
Material: 25/55-55
Frequency: 1.59Hz
Temperature: 0°C

laboratory	value 1 [kPa]	value 2 [kPa]	mean value [kPa]	deviation [%]	lab deviation [%]	z score
17	63 851	60 026	61 938	3,09	-28,90	-1,24
13	61 263	68 540	64 901	5,61	-25,50	-1,09
1	67 608	70 008	68 808	1,74	-21,01	-0,90
2	70 383	73 462	71 922	2,14	-17,44	-0,75
6	76 290	81 150	78 720	3,09	-9,63	-0,41
12	76 941	80 732	78 837	2,40	-9,50	-0,41
19	89 680	73 022	81 351	10,24	-6,61	-0,28
20	82 160	80 835	81 498	0,81	-6,44	-0,28
5	87 396	82 521	84 959	2,87	-2,47	-0,11
14	86 368	84 556	85 462	1,06	-1,89	-0,08
9	83 678	87 871	85 775	2,44	-1,53	-0,07
4	90 105	86 018	88 062	2,32	1,09	0,05
3	83 337	95 259	89 298	6,68	2,51	0,11
21	92 800	86 500	89 650	3,51	2,91	0,13
18	91 181	88 279	89 730	1,62	3,01	0,13
7	91 160	92 910	92 035	0,95	5,65	0,24
10	101 030	100 310	100 670	0,36	15,57	0,67
8	102 230	102 510	102 370	0,14	17,52	0,75
16	108 275	100 928	104 601	3,51	20,08	0,86
22	98 625	114 288	106 456	7,36	22,21	0,95
11	120 630	123 951	122 291	1,36	40,38	1,73
15	159 570	153 020	156 295	2,10	79,42	3,41
		general mean:	87 111			

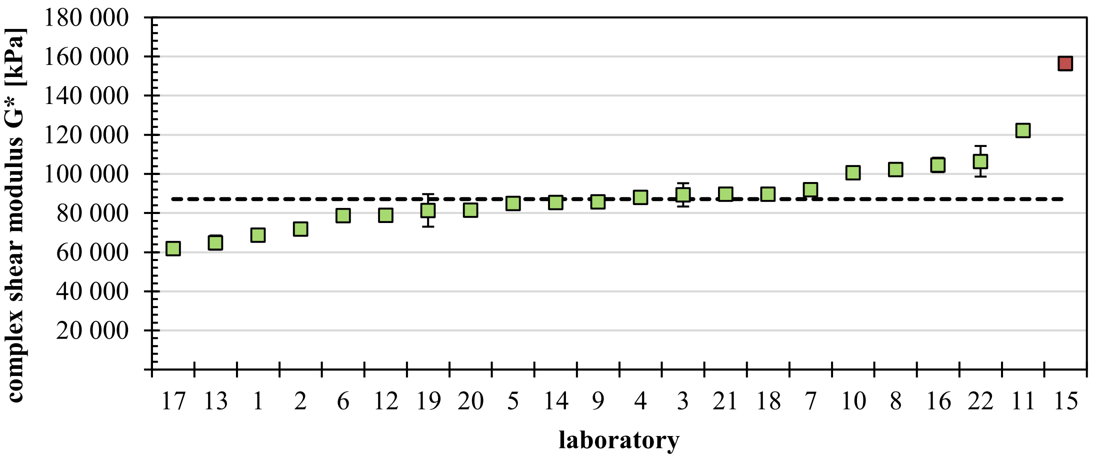

Figure 8: complex shear modulus G* of polymer modified binder 25/55-55 at 1.59Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.5.2 Phase angle δ

Characteristic: phase angle δ
Material: 50/70
Frequency: 1.59Hz
Temperature: -30°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[°]	[°]	[°]	[%]	[%]	
7	4.83	4.74	4.79	1.02	-30.44	-1.79
10	5.07	5.12	5.10	0.49	-25.94	-1.53
11	5.63	5.58	5.61	0.45	-18.50	-1.09
8	5.87	5.59	5.73	2.44	-16.71	-0.98
16	6.16	6.42	6.29	2.13	-8.56	-0.50
19	6.52	6.52	6.52	0.00	-5.18	-0.31
4	5.55	8.04	6.80	18.32	-1.23	-0.07
22	6.96	6.83	6.89	0.94	0.19	0.01
3	7.00	6.80	6.90	1.45	0.30	0.02
13	8.48	5.57	7.03	20.73	2.14	0.13
21	7.09	7.23	7.16	0.97	4.06	0.24
9	7.30	7.20	7.25	0.69	5.39	0.32
12	7.31	7.45	7.38	0.95	7.28	0.43
14	7.58	7.48	7.53	0.66	9.46	0.56
20	7.52	7.66	7.59	0.96	10.35	0.61
17	9.27	8.82	9.04	2.46	31.48	1.85
6	9.50	9.20	9.35	1.60	35.91	2.11
		general mean:	6.88			

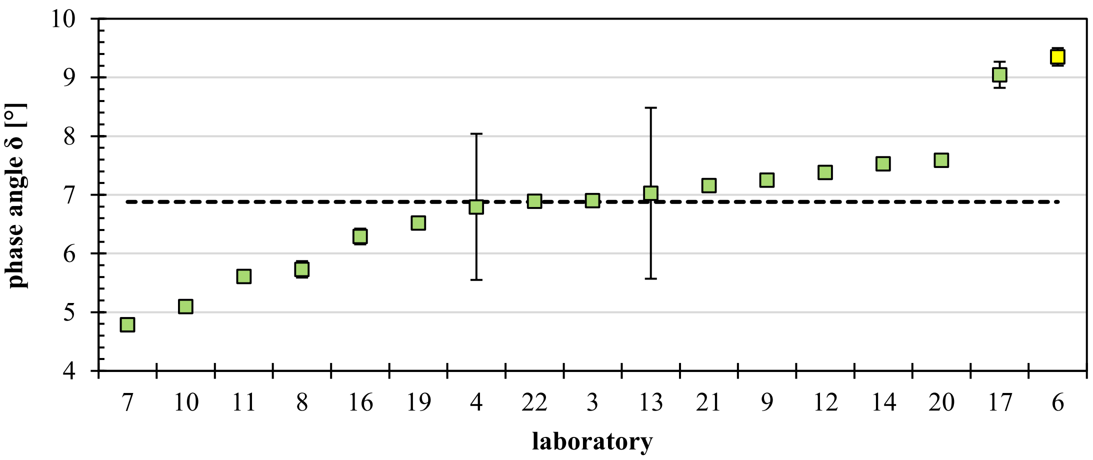

Figure 9: Phase angle δ of plain binder 50/70 at 1.59Hz and-30°C

Lab 1, 2, 5, 15 and 18 were not able to test at test temperature -30°C.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 50/70
Frequency: 1.59Hz
Temperature: -20°C

laboratory	value 1 [°]	value 2 [°]	mean value [°]	deviation [%]	lab deviation [%]	z score
4	8.69	9.66	9.18	5.29	-17.72	-1.41
16	9.36	9.76	9.56	2.06	-14.26	-1.13
11	10.15	9.34	9.74	4.17	-12.63	-1.00
10	9.82	9.90	9.86	0.41	-11.58	-0.92
1	9.40	10.52	9.96	5.62	-10.69	-0.85
7	10.03	10.04	10.04	0.05	-10.01	-0.79
18	10.86	9.44	10.15	7.00	-8.98	-0.71
19	10.38	10.34	10.36	0.19	-7.09	-0.56
3	11.00	10.70	10.85	1.38	-2.70	-0.21
8	11.07	10.63	10.85	2.03	-2.70	-0.21
2	10.91	11.21	11.06	1.37	-0.85	-0.07
20	11.30	11.11	11.20	0.83	0.47	0.04
12	11.39	11.51	11.45	0.52	2.68	0.21
14	11.60	11.49	11.55	0.48	3.53	0.28
13	12.47	10.81	11.64	7.14	4.39	0.35
22	12.21	12.14	12.18	0.29	9.20	0.73
21	12.30	12.17	12.24	0.53	9.73	0.77
9	12.90	12.80	12.85	0.39	15.23	1.21
17	14.21	12.53	13.37	6.29	19.91	1.58
6	15.20	14.70	14.95	1.67	34.06	2.70
		general mean:	11.15			

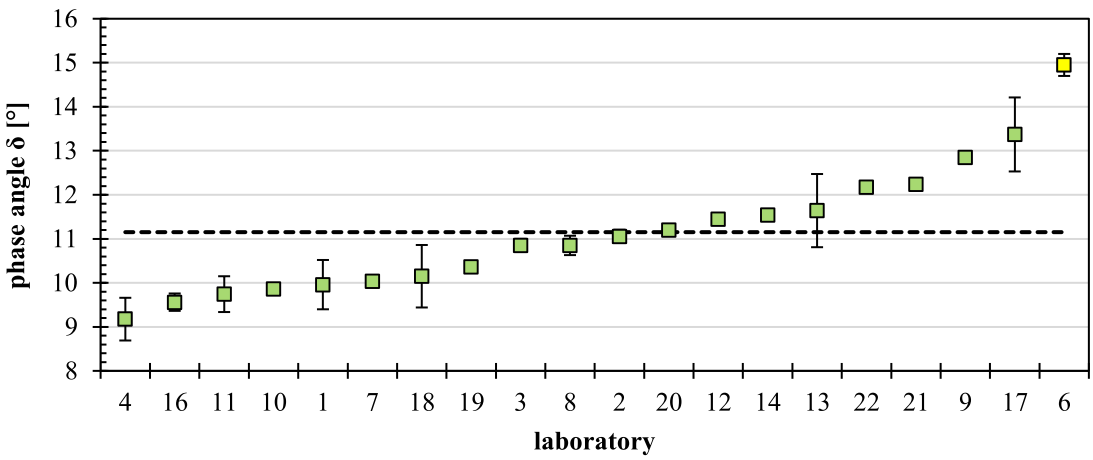

Figure 10: Phase angle δ of plain binder 50/70 at 1.59Hz and-20°C

Lab 5 and 15 were not able to test at test temperature -20°C.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 50/70
Frequency: 1.59Hz
Temperature: -10°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[°]	[°]	[°]	[%]	[%]	
4	17.18	17.17	17.18	0.03	-16.83	-2.02
16	18.18	18.71	18.45	1.43	-10.66	-1.28
18	17.94	19.54	18.74	4.27	-9.25	-1.11
11	19.95	17.77	18.86	5.79	-8.67	-1.04
3	19.40	19.20	19.30	0.52	-6.53	-0.79
10	19.94	20.05	20.00	0.28	-3.17	-0.38
19	19.00	21.12	20.06	5.27	-2.85	-0.34
20	20.17	20.09	20.13	0.20	-2.52	-0.30
7	20.23	20.16	20.20	0.17	-2.20	-0.26
14	20.14	20.32	20.23	0.44	-2.03	-0.24
2	20.34	20.23	20.29	0.27	-1.75	-0.21
12	20.60	20.55	20.58	0.12	-0.36	-0.04
9	20.90	20.80	20.85	0.24	0.97	0.12
15	21.19	20.60	20.90	1.41	1.19	0.14
8	21.59	21.26	21.43	0.77	3.76	0.45
13	22.00	21.85	21.92	0.34	6.17	0.74
21	21.93	21.97	21.95	0.10	6.30	0.76
22	22.37	21.84	22.10	1.19	7.04	0.85
1	22.07	22.62	22.34	1.23	8.19	0.98
6	23.30	22.70	23.00	1.30	11.38	1.37
17	25.18	25.13	25.16	0.09	21.83	2.62
		general mean:	20.65			

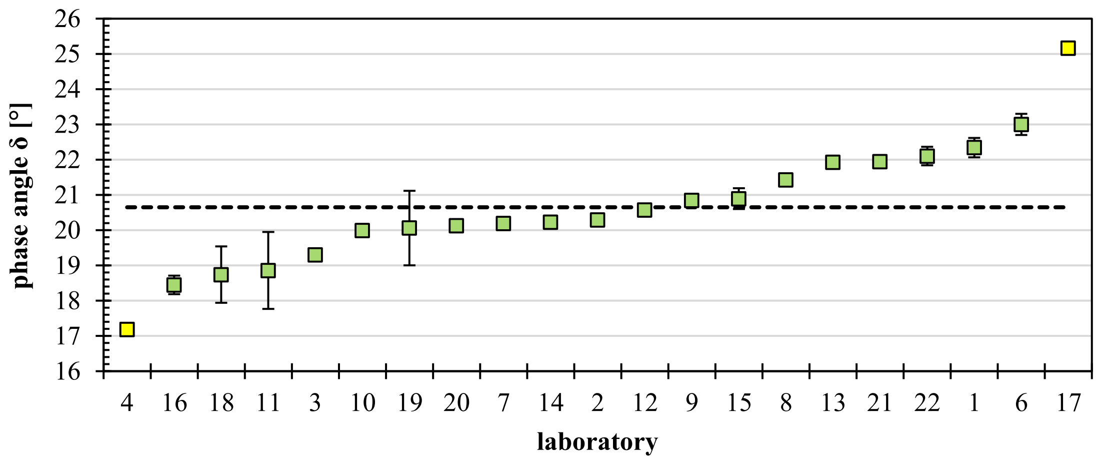

Figure 11: Phase angle δ of plain binder 50/70 at 1.59Hz and-10°C

Lab 5 was not able to test at test temperature -10°C.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 50/70
Frequency: 1.59Hz
Temperature: 0°C

laboratory	value 1 [°]	value 2 [°]	mean value [°]	deviation [%]	lab deviation [%]	z score
11	31.62	28.72	30.17	4.80	-9.83	-1.70
4	30.37	30.36	30.37	0.02	-9.26	-1.60
14	31.24	31.72	31.48	0.76	-5.93	-1.02
16	31.34	32.49	31.91	1.80	-4.63	-0.80
3	32.30	32.00	32.15	0.47	-3.93	-0.68
20	32.17	32.13	32.15	0.05	-3.92	-0.68
2	32.42	32.05	32.23	0.58	-3.68	-0.64
7	32.63	32.47	32.55	0.25	-2.73	-0.47
10	32.63	32.71	32.67	0.12	-2.37	-0.41
9	33.20	32.50	32.85	1.07	-1.84	-0.32
12	33.03	32.80	32.92	0.35	-1.64	-0.28
19	33.05	33.24	33.14	0.29	-0.96	-0.17
15	33.29	33.10	33.20	0.29	-0.80	-0.14
18	34.23	33.22	33.73	1.50	0.78	0.13
22	34.49	33.63	34.06	1.26	1.78	0.31
5	34.43	34.16	34.29	0.39	2.48	0.43
8	34.50	34.29	34.40	0.31	2.78	0.48
6	35.90	35.10	35.50	1.13	6.08	1.05
21	35.40	35.67	35.54	0.37	6.19	1.07
1	35.99	36.63	36.31	0.89	8.50	1.47
13	36.91	36.59	36.75	0.44	9.82	1.70
17	37.80	37.91	37.85	0.15	13.12	2.26
		general mean:	33.46			

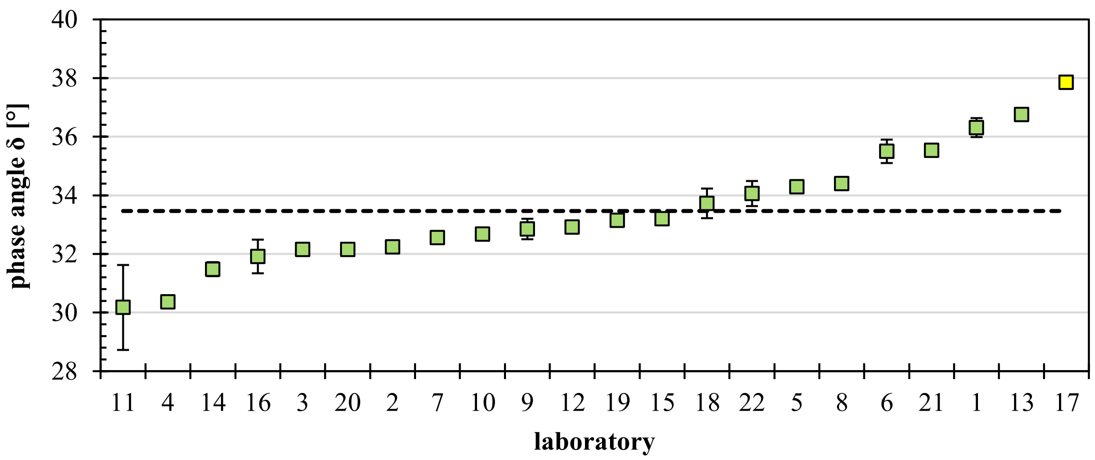

Figure 12: Phase angle δ of plain binder 50/70 at 1.59Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 25/55-55
Frequency: 1.59Hz
Temperature: -30°C

laboratory	value 1 [°]	value 2 [°]	mean value [°]	deviation [%]	lab deviation [%]	z score
7	4.69	4.59	4.64	1.12	-30.66	-1.75
5	5.04	-	5.04	0.00	-24.72	-1.41
10	5.32	5.24	5.28	0.76	-21.09	-1.20
11	5.43	5.42	5.43	0.10	-18.87	-1.07
8	5.70	5.38	5.54	2.89	-17.21	-0.98
16	5.71	5.86	5.78	1.30	-13.56	-0.77
3	6.07	6.32	6.19	2.01	-7.44	-0.42
19	6.64	6.41	6.52	1.72	-2.49	-0.14
13	7.56	5.74	6.65	13.67	-0.64	-0.04
9	6.90	6.90	6.90	0.00	3.12	0.18
22	6.86	6.95	6.90	0.60	3.17	0.18
12	6.99	6.93	6.96	0.43	4.01	0.23
21	7.00	7.07	7.03	0.53	5.14	0.29
14	7.40	7.39	7.40	0.07	10.52	0.60
20	7.49	7.32	7.40	1.15	10.64	0.61
4	7.55	7.67	7.61	0.79	13.73	0.78
6	8.50	8.40	8.45	0.59	26.28	1.50
17	8.35	8.97	8.66	3.59	29.37	1.67
18	10.11	7.38	8.75	15.61	30.69	1.75
		general mean:	6.69			

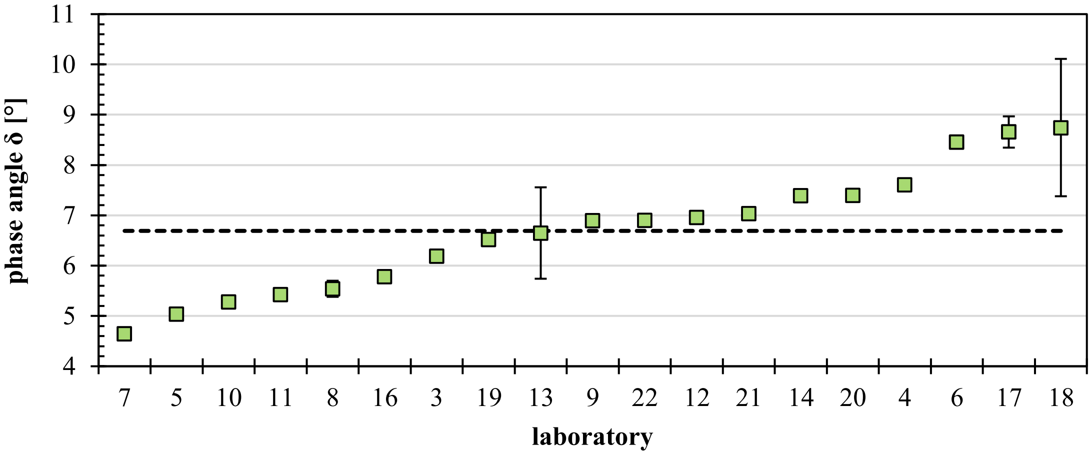

Figure 13: Phase angle δ polymer modified binder 25/55-55 at 1.59Hz and-30°C

Lab 1. 2 and 15 were not able to test at test temperature -30°C.

Lab 5 only provided one test result.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 25/55-55
Frequency: 1.59Hz
Temperature: -20°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[°]	[°]	[°]	[%]	[%]	
16	8.26	8.49	8.38	1.38	-18.59	-1.68
7	8.81	8.88	8.84	0.36	-14.04	-1.27
11	9.08	8.95	9.02	0.70	-12.34	-1.11
5	9.04	-	9.04	0.00	-12.11	-1.09
10	9.15	9.08	9.12	0.38	-11.40	-1.03
3	9.21	9.49	9.35	1.54	-9.13	-0.82
8	9.85	9.44	9.65	2.13	-6.25	-0.56
2	10.29	9.52	9.91	3.93	-3.73	-0.34
1	10.08	9.97	10.03	0.51	-2.55	-0.23
18	10.13	10.17	10.15	0.20	-1.34	-0.12
19	10.32	10.37	10.35	0.22	0.57	0.05
12	10.46	10.37	10.42	0.43	1.23	0.11
4	10.52	10.47	10.50	0.24	2.01	0.18
14	10.69	10.94	10.82	1.16	5.12	0.46
20	10.71	10.98	10.85	1.26	5.42	0.49
21	10.85	10.89	10.87	0.19	5.66	0.51
13	11.43	10.53	10.98	4.10	6.72	0.61
22	11.06	11.15	11.10	0.38	7.91	0.71
9	11.20	11.40	11.30	0.88	9.83	0.89
17	11.95	12.28	12.11	1.35	17.74	1.60
6	13.50	13.10	13.30	1.50	29.27	2.64
		general mean:	10.29			

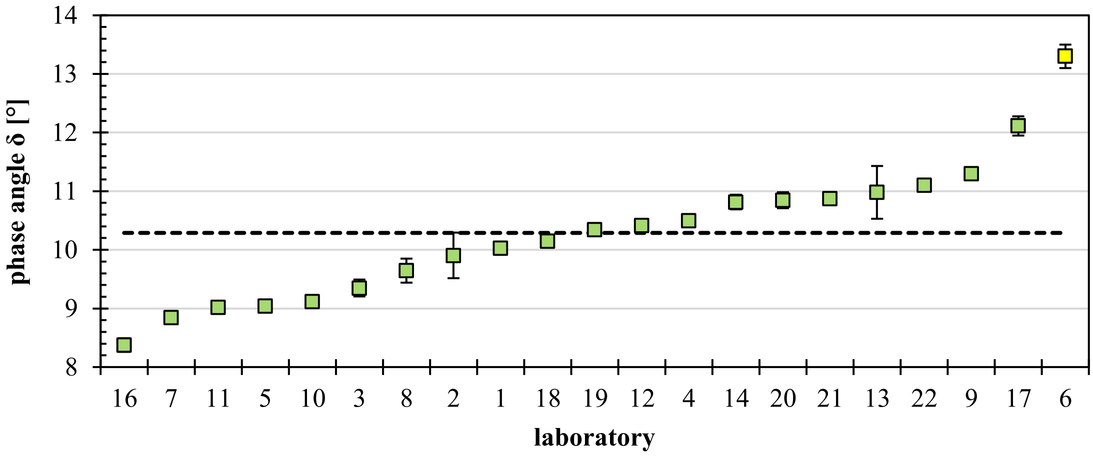

Figure 14: Phase angle δ polymer modified binder 25/55-55 at 1.59Hz and-20°C

Lab 15 was not able to test at test temperature -20°C.

Lab 5 only provided one test result.

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 25/55-55
Frequency: 1.59Hz
Temperature: -10°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[°]	[°]	[°]	[%]	[%]	
16	15.46	15.92	15.69	1.47	-15.12	-2.25
3	16.48	16.92	16.70	1.32	-9.69	-1.44
4	16.97	17.08	17.03	0.32	-7.92	-1.18
11	17.46	17.19	17.32	0.79	-6.30	-0.94
7	17.70	17.61	17.66	0.25	-4.52	-0.67
2	18.02	17.49	17.75	1.49	-3.99	-0.59
10	17.75	17.82	17.79	0.20	-3.81	-0.57
18	18.17	18.14	18.16	0.08	-1.81	-0.27
5	18.07	18.40	18.23	0.93	-1.39	-0.21
12	18.41	18.36	18.39	0.14	-0.57	-0.08
14	18.27	18.72	18.50	1.22	0.03	0.00
9	18.60	18.50	18.55	0.27	0.33	0.05
15	18.56	18.59	18.58	0.08	0.46	0.07
8	18.84	18.32	18.58	1.40	0.49	0.07
1	18.80	18.43	18.62	0.98	0.68	0.10
21	18.94	18.83	18.88	0.28	2.12	0.32
20	19.58	18.92	19.25	1.70	4.11	0.61
22	19.63	19.50	19.56	0.33	5.79	0.86
19	19.36	20.60	19.98	3.10	8.06	1.20
6	20.30	19.90	20.10	1.00	8.71	1.30
13	20.37	19.94	20.15	1.07	9.00	1.34
17	21.36	21.29	21.33	0.17	15.34	2.28
		general mean:	18.49			

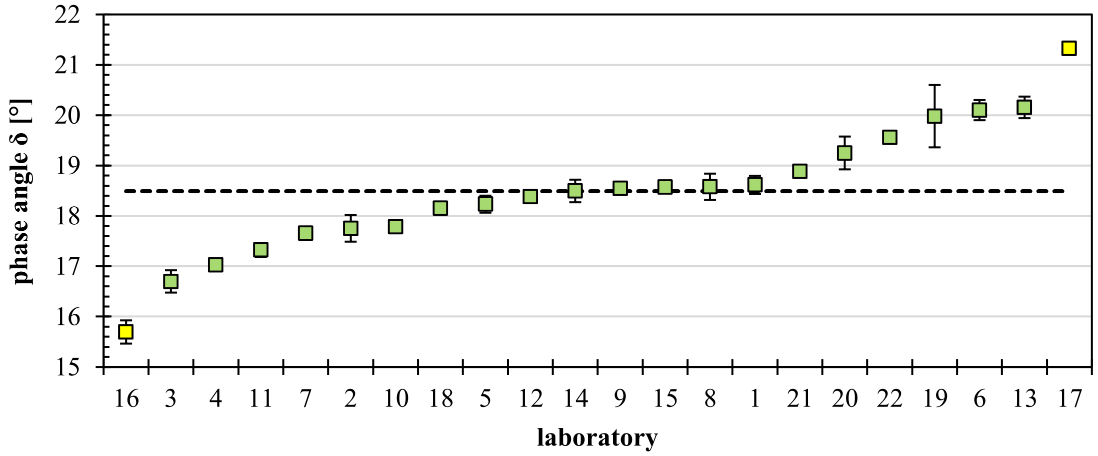

Figure 15: Phase angle δ polymer modified binder 25/55-55 at 1.59Hz and-10°C

https://doi.org/10.24355/dbbs.084-202111031047-0

Characteristic: phase angle δ
Material: 25/55-55
Frequency: 1.59Hz
Temperature: 0°C

laboratory	value 1	value 2	mean value	deviation	lab deviation	z score
	[°]	[°]	[°]	[%]	[%]	
16	27.43	28.39	27.91	1.72	-6.35	-1.49
11	28.28	27.76	28.02	0.93	-6.00	-1.40
4	28.11	28.15	28.13	0.07	-5.62	-1.32
3	28.34	28.76	28.55	0.73	-4.21	-0.99
2	29.16	28.61	28.88	0.94	-3.09	-0.72
7	29.30	29.27	29.29	0.05	-1.74	-0.41
9	29.10	29.50	29.30	0.68	-1.69	-0.40
10	29.27	29.43	29.35	0.27	-1.52	-0.36
14	29.08	29.62	29.35	0.92	-1.52	-0.36
21	29.33	29.41	29.37	0.14	-1.46	-0.34
18	29.31	29.46	29.39	0.26	-1.41	-0.33
5	29.29	29.82	29.55	0.88	-0.84	-0.20
20	29.87	29.48	29.67	0.66	-0.44	-0.10
12	29.76	29.62	29.69	0.24	-0.38	-0.09
15	30.14	30.14	30.14	0.00	1.13	0.26
8	30.56	30.11	30.34	0.74	1.78	0.42
22	30.58	30.35	30.46	0.38	2.21	0.52
1	29.78	31.44	30.61	2.71	2.70	0.63
17	31.06	31.11	31.09	0.08	4.31	1.01
6	31.70	31.30	31.50	0.63	5.69	1.33
19	30.57	33.08	31.83	3.95	6.78	1.59
13	33.51	33.07	33.29	0.66	11.69	2.74
		general mean:	29.80			

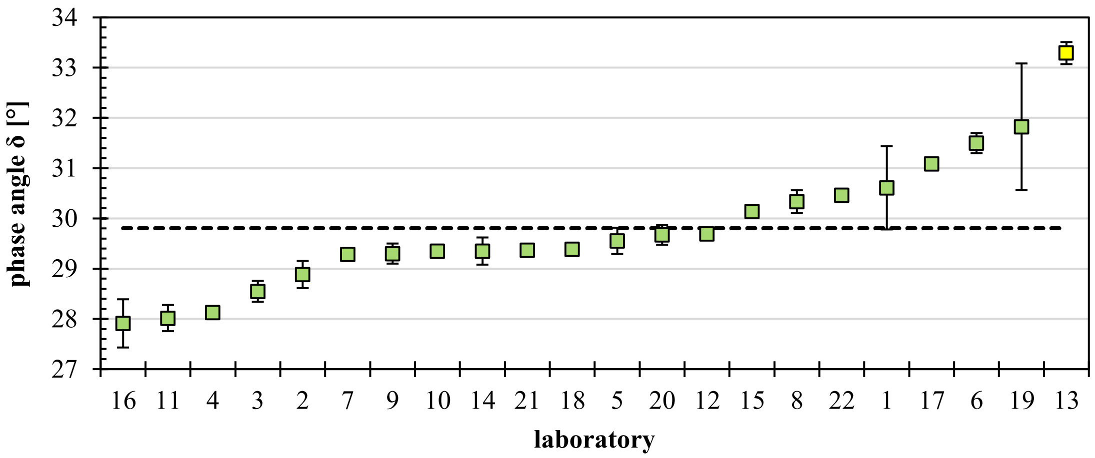

Figure 16: Phase angle δ polymer modified binder 25/55-55 at 1.59Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.6 Graphical analysis of rheological parameters @0.1Hz

6.6.1 Complex Shear Modulus G*

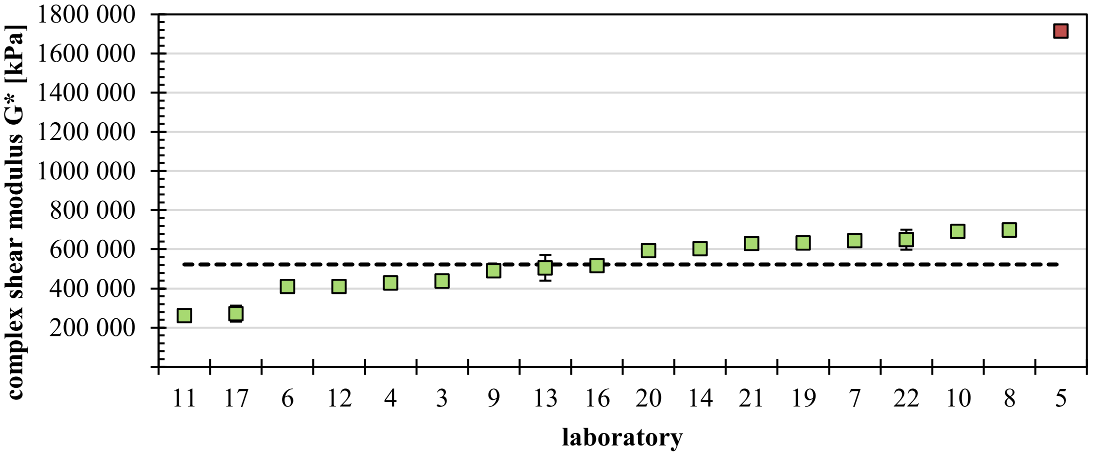

Figure 17: complex shear modulus G* of plain binder 50/70 at 0.1Hz and -30°C

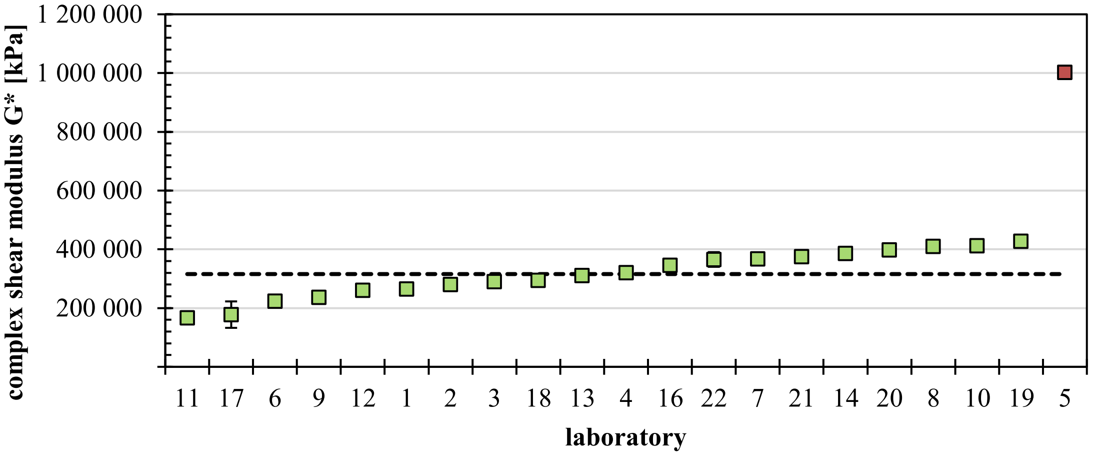

Figure 18: complex shear modulus G* of plain binder 50/70 at 0.1Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

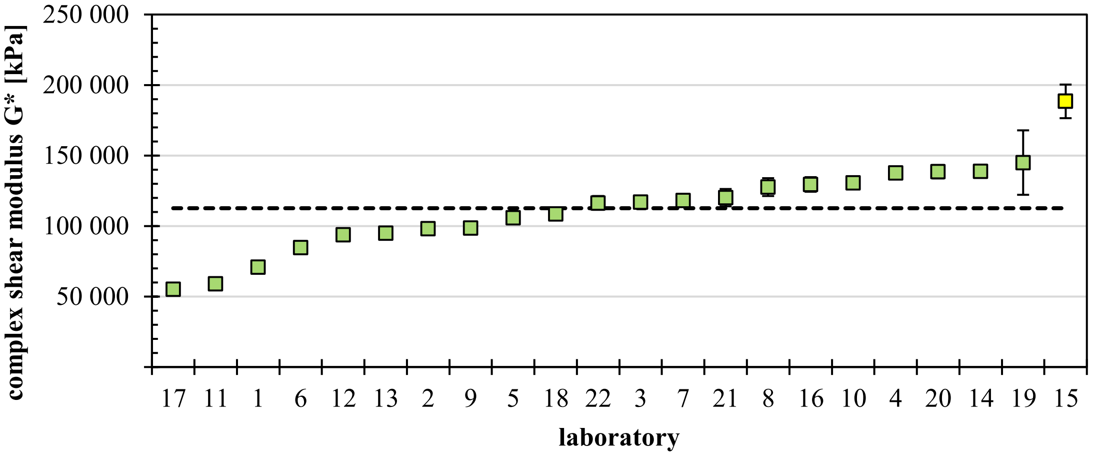

Figure 19: complex shear modulus G* of plain binder 50/70 at 0.1Hz and -10°C

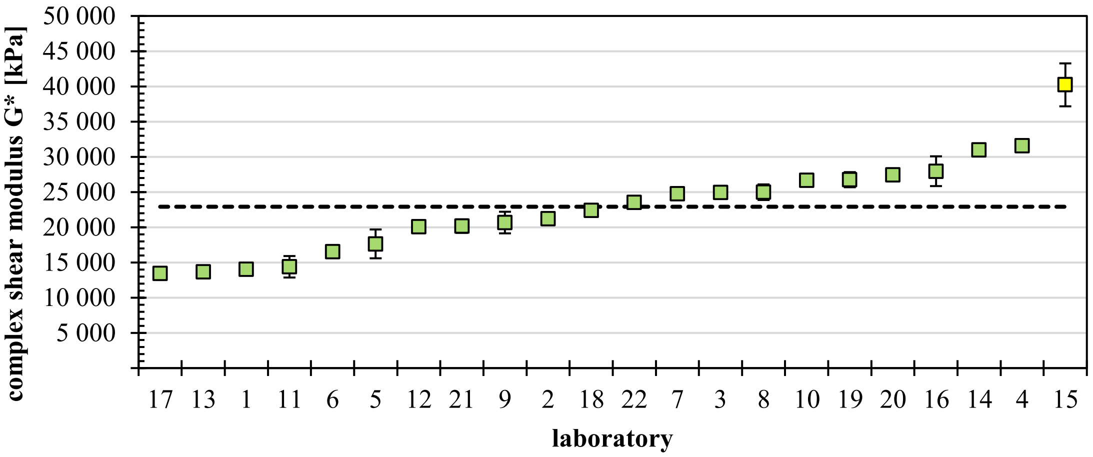

Figure 20: complex shear modulus G* of plain binder 50/70 at 0.1Hz and 0°C

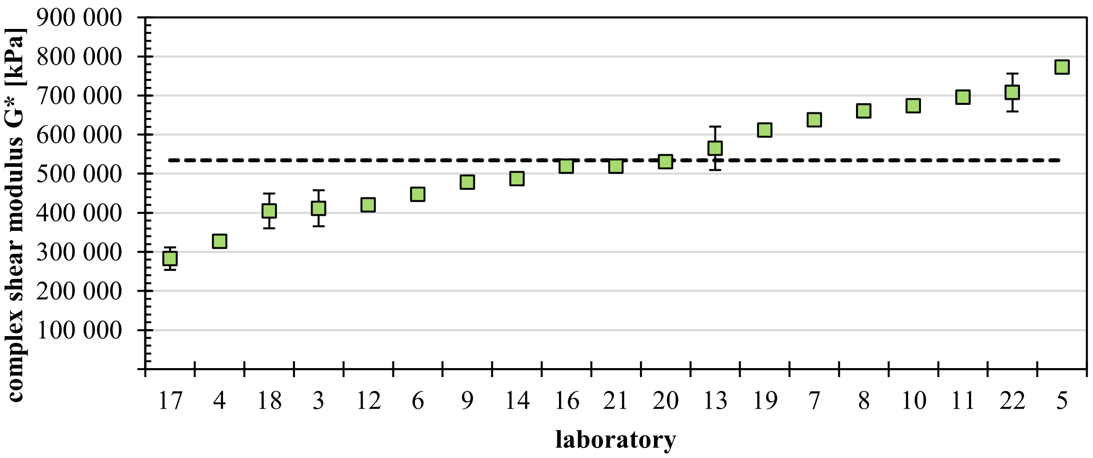

Figure 21: complex shear modulus G* of polymer modified binder 25/55-55 at 0.1Hz and -30°C

https://doi.org/10.24355/dbbs.084-202111031047-0

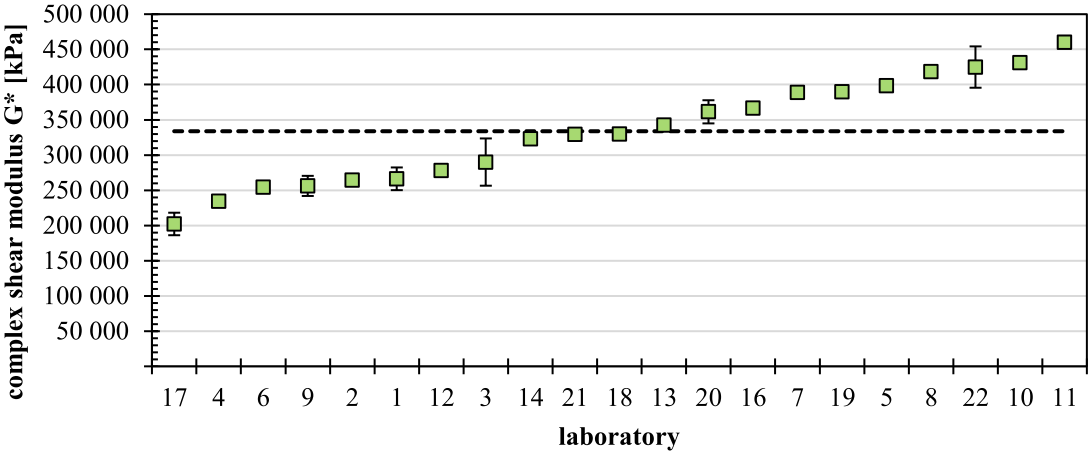

Figure 22: complex shear modulus G* of polymer modified binder 25/55-55 at 0.1Hz and -20°C

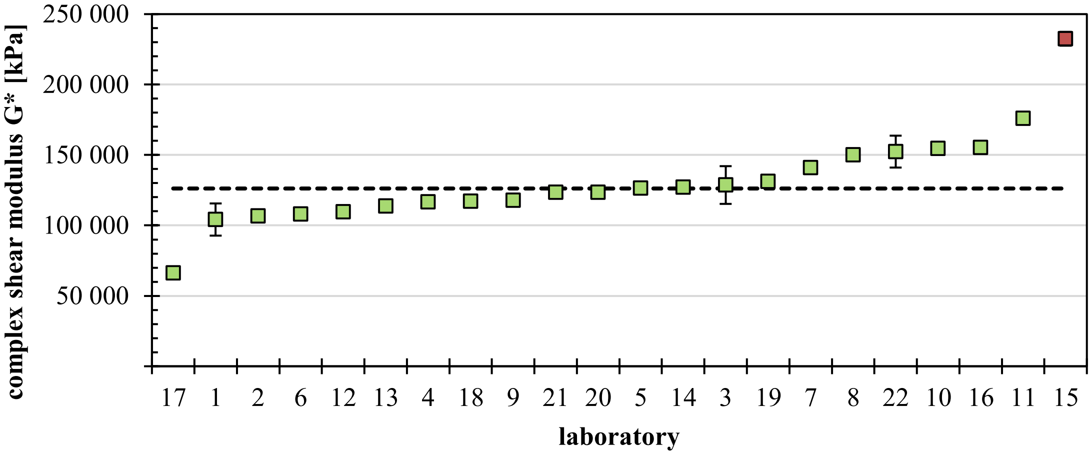

Figure 23: complex shear modulus G* of polymer modified binder 25/55-55 at 0.1Hz and -10°C

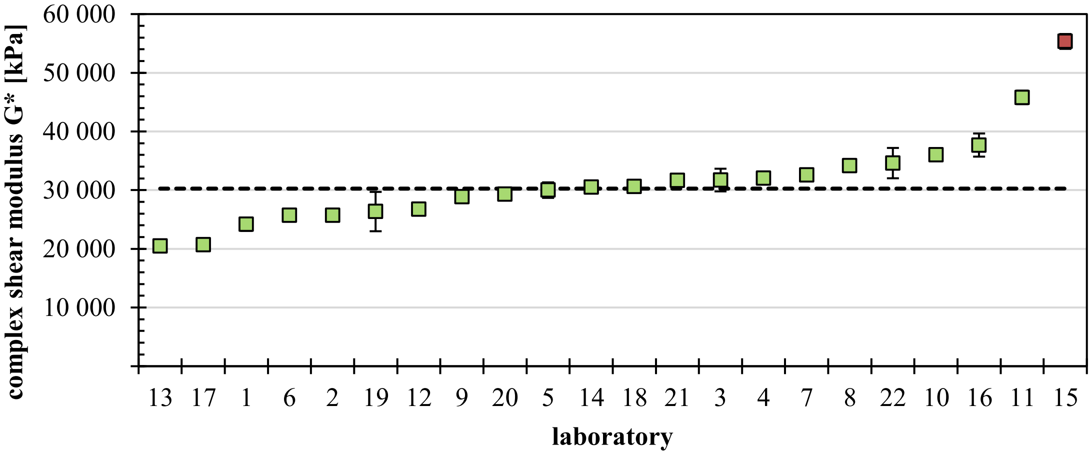

Figure 24: complex shear modulus G* of polymer modified binder 25/55-55 at 0.1Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.6.2 Phase Angle δ

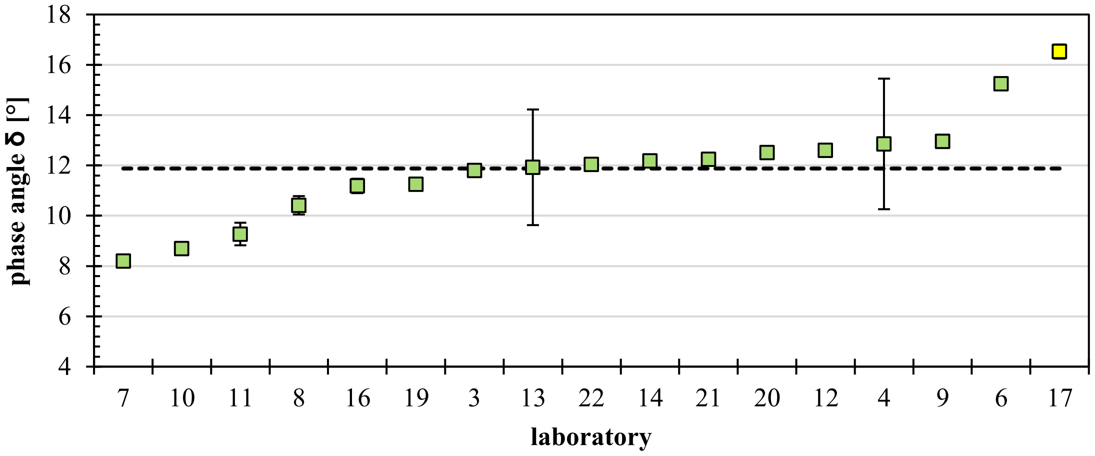

Figure 25: Phase angle δ of plain binder 50/70 at 0.1Hz and -30°C

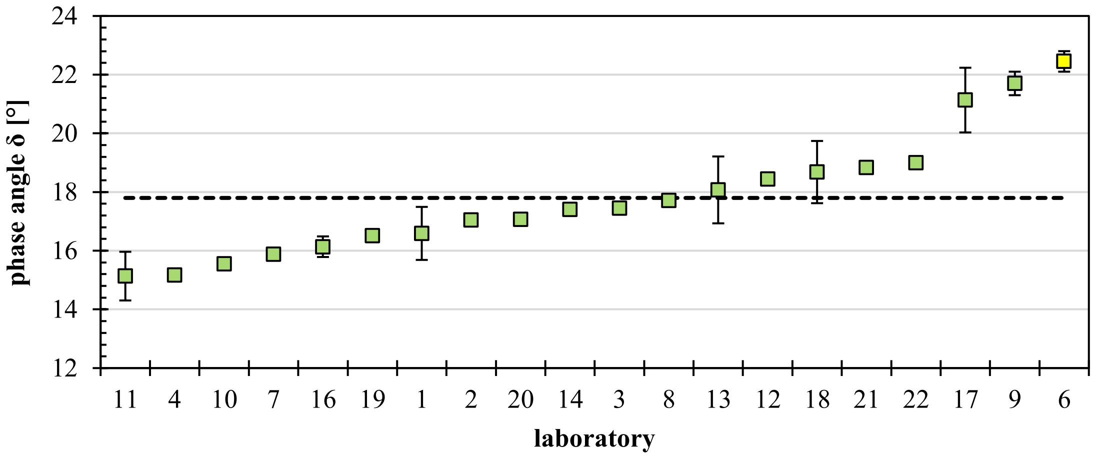

Figure 26: Phase angle δ of plain binder 50/70 at 0.1Hz and -20°C

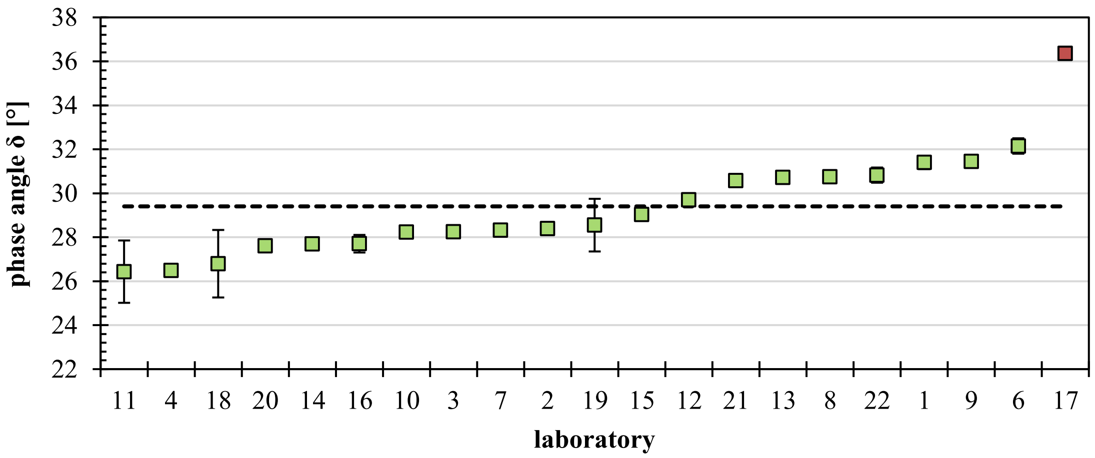

Figure 27: Phase angle δ of plain binder 50/70 at 0.1Hz and -10°C

https://doi.org/10.24355/dbbs.084-202111031047-0

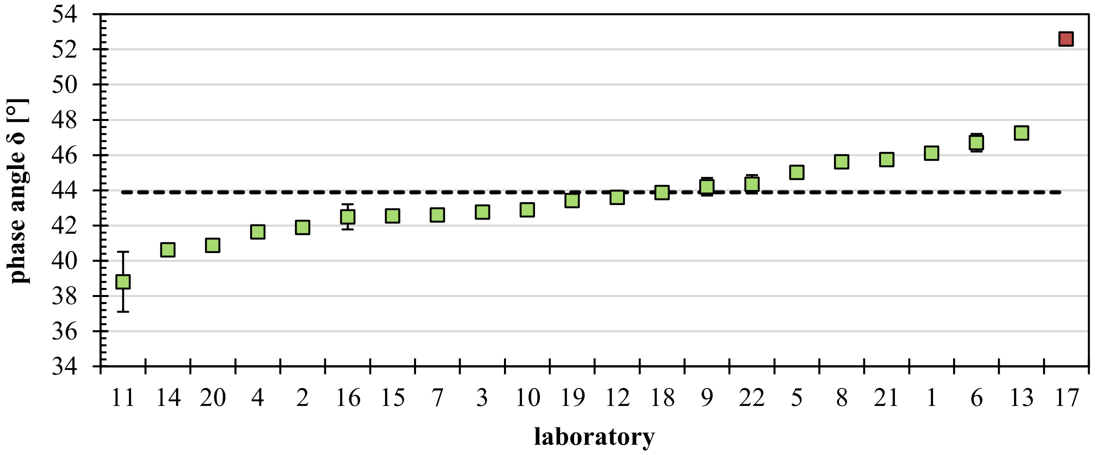

Figure 28: Phase angle δ of plain binder 50/70 at 0.1Hz and 0°C

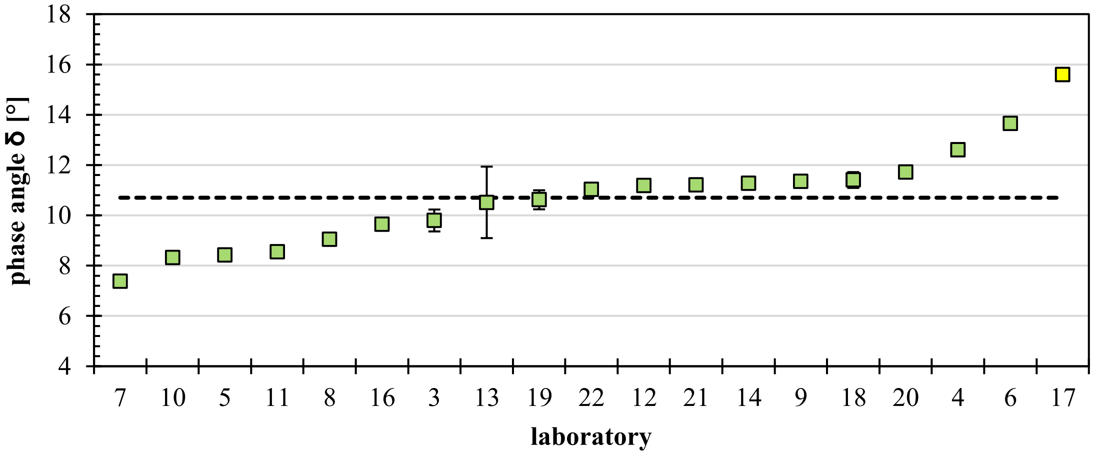

Figure 29: Phase angle δ of polymer modified binder 25/55-55 at 0.1Hz and -30°C

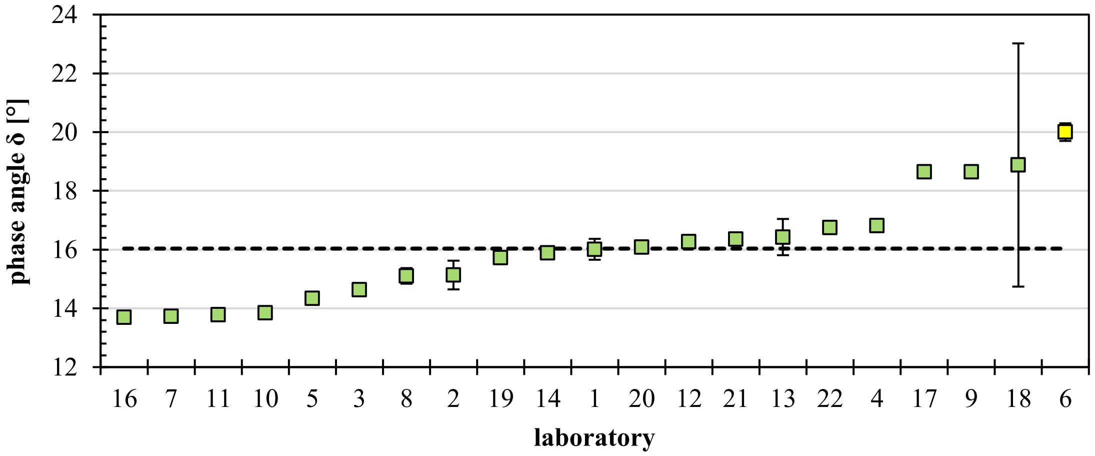

Figure 30: Phase angle δ of polymer modified binder 25/55-55 at 0.1Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

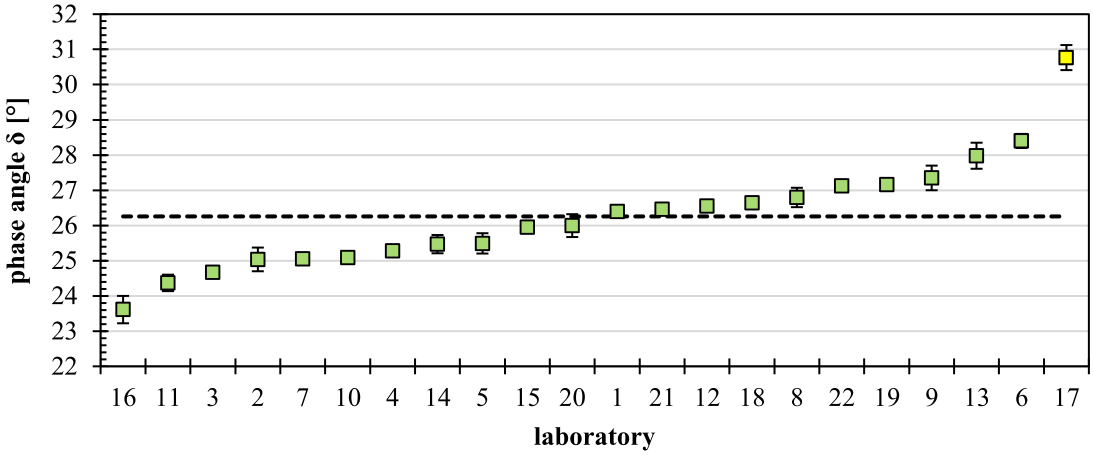

Figure 31: Phase angle δ of polymer modified binder 25/55-55 at 0.1Hz and -10°C

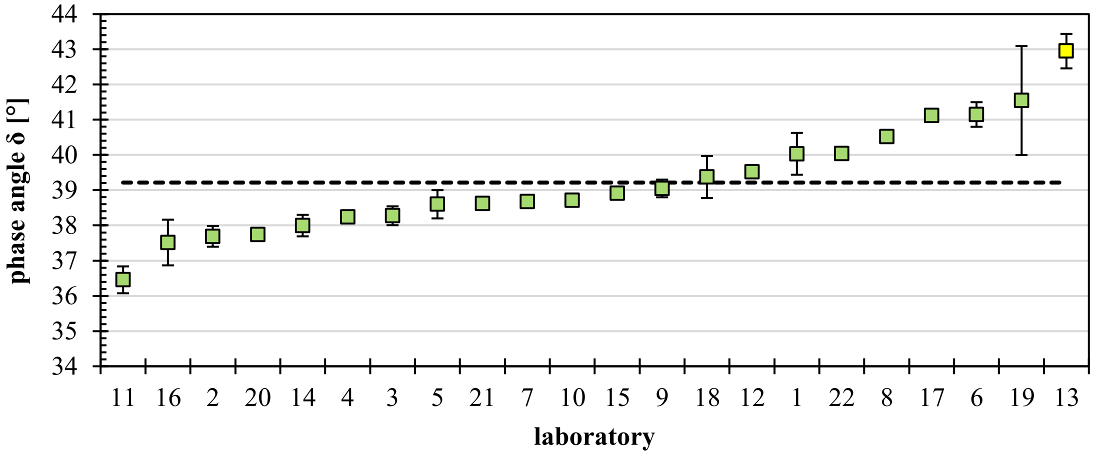

Figure 32: Phase angle δ of polymer modified binder 25/55-55 at 0.1Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.7 Graphical analysis of rheological parameters @1.0Hz

6.7.1 Complex Shear Modulus G*

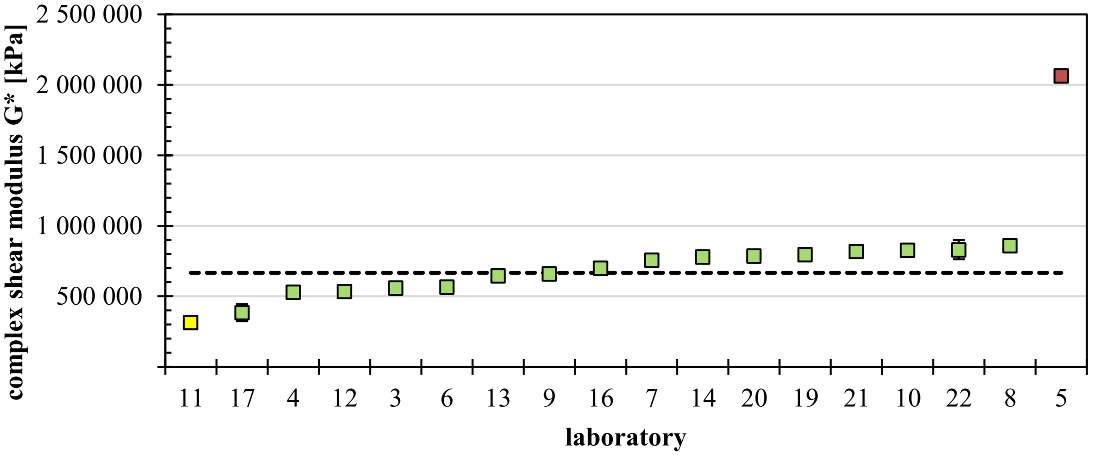

Figure 33: complex shear modulus G* of plain binder 50/70 at 1.0Hz and -30°C

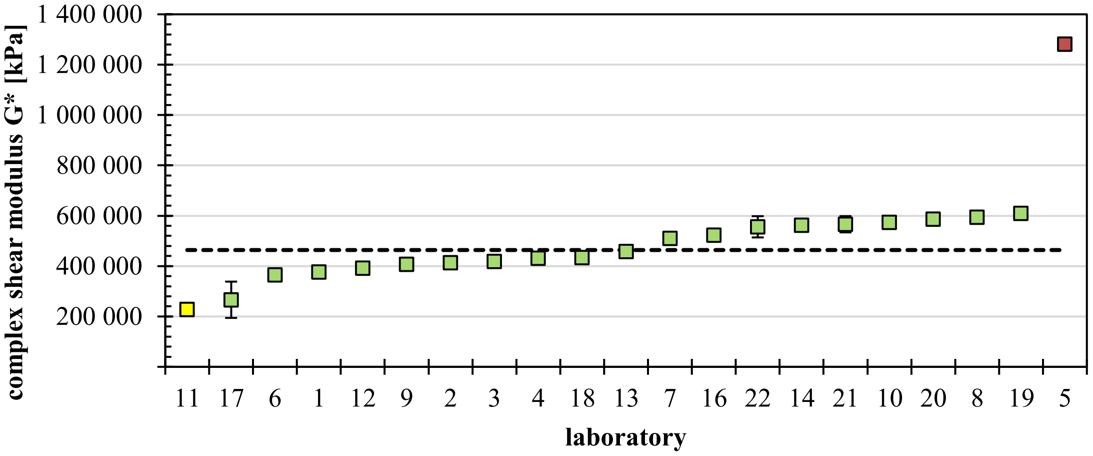

Figure 34: complex shear modulus G* of plain binder 50/70 at 1.0Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

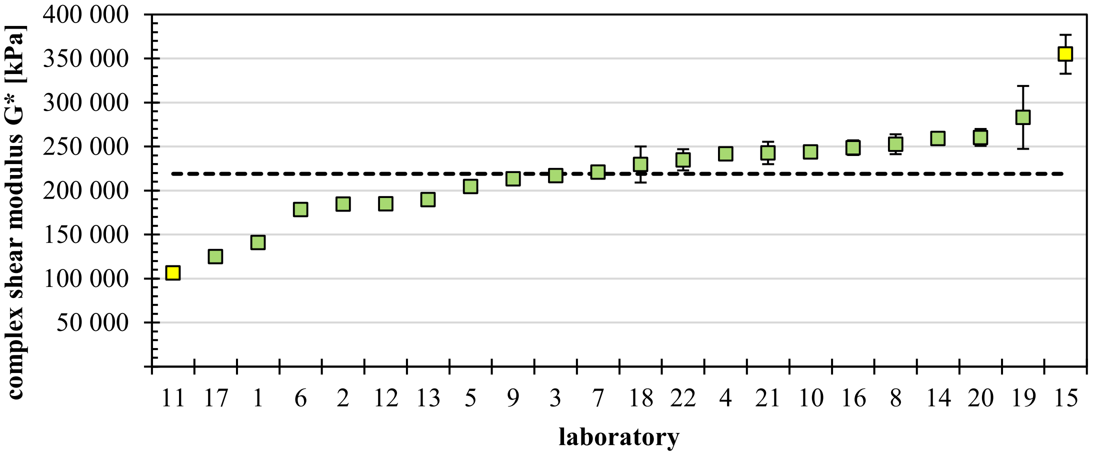

Figure 35: complex shear modulus G* of plain binder 50/70 at 1.0Hz and -10°C

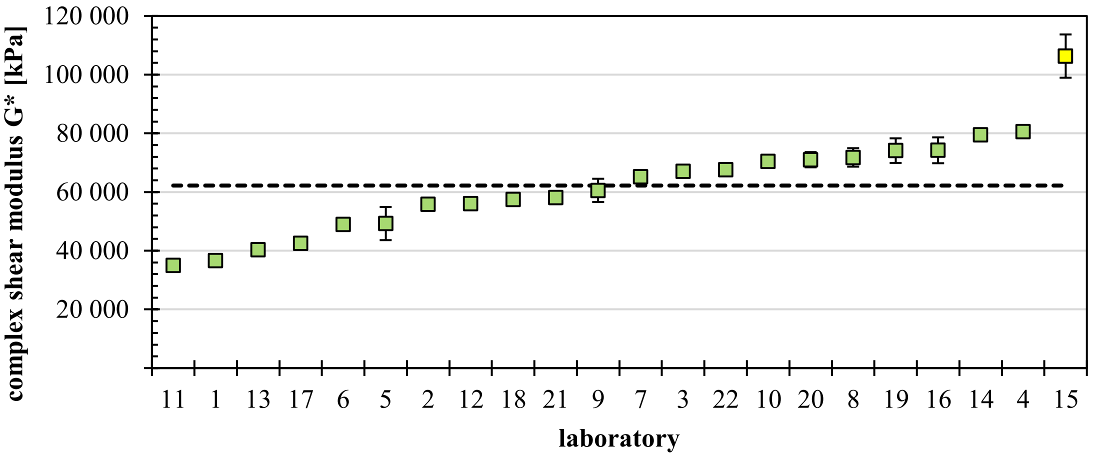

Figure 36: complex shear modulus G* of plain binder 50/70 at 1.0Hz and 0°C

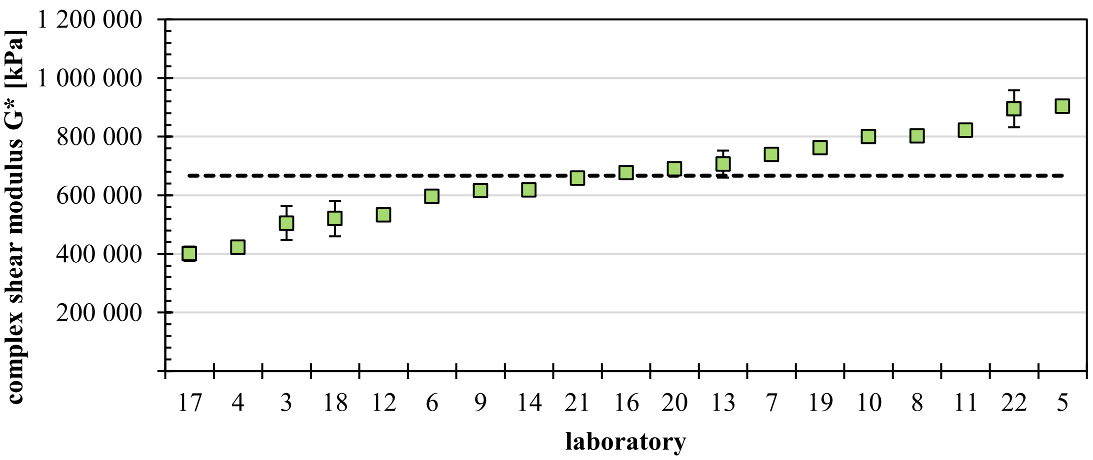

Figure 37: complex shear modulus G* of polymer modified binder 25/55-55 at 1.0Hz and -30°C

https://doi.org/10.24355/dbbs.084-202111031047-0

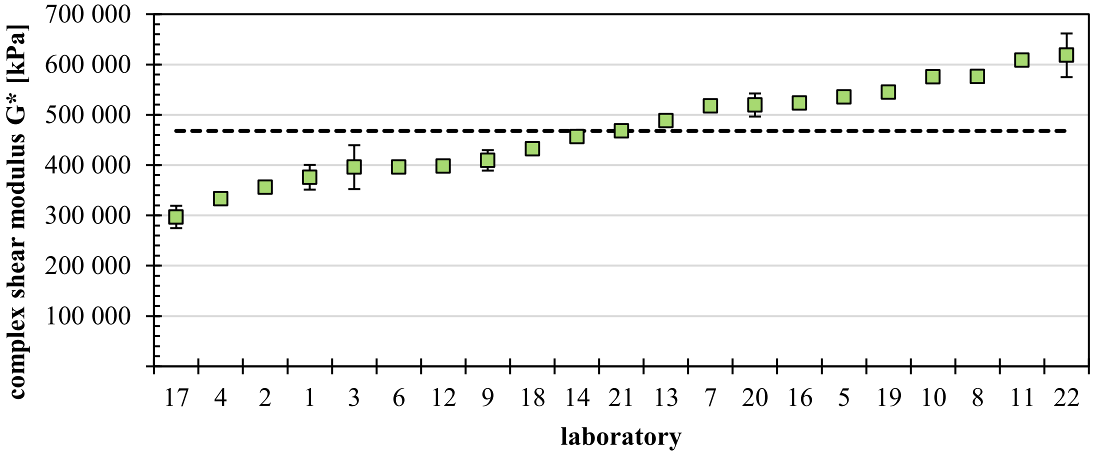

Figure 38: complex shear modulus G* of polymer modified binder 25/55-55 at 1.0Hz and -20°C

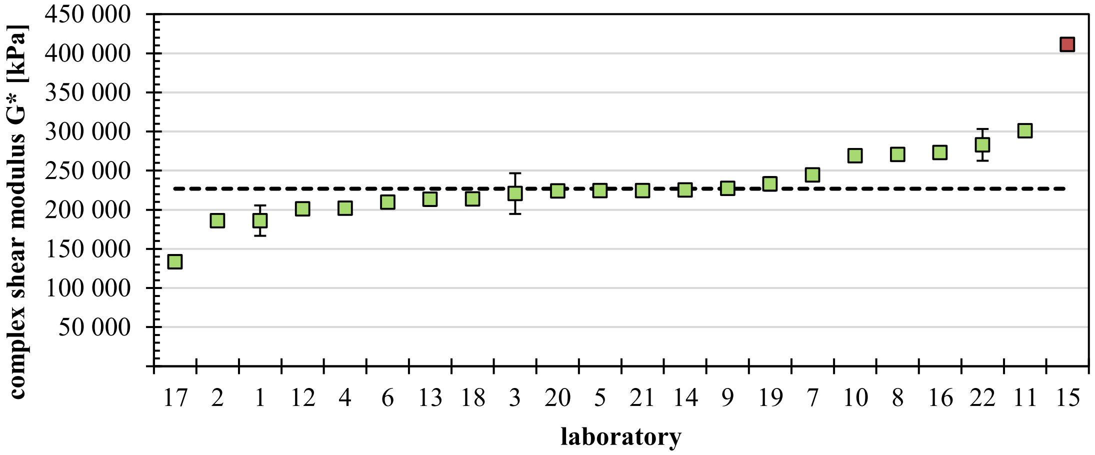

Figure 39: complex shear modulus G* of polymer modified binder 25/55-55 at 1.0Hz and -10°C

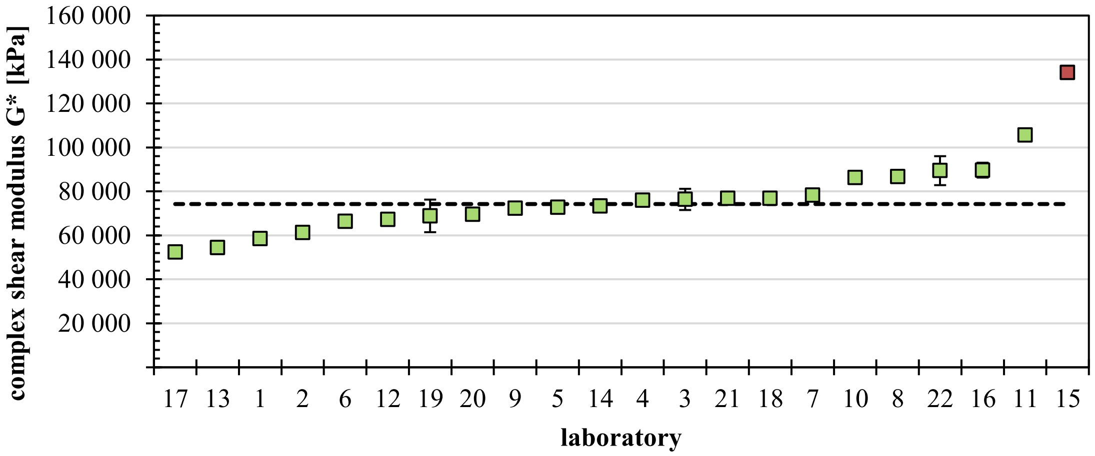

Figure 40: complex shear modulus G* of polymer modified binder 25/55-55 at 1.0Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.7.2 Phase Angle δ

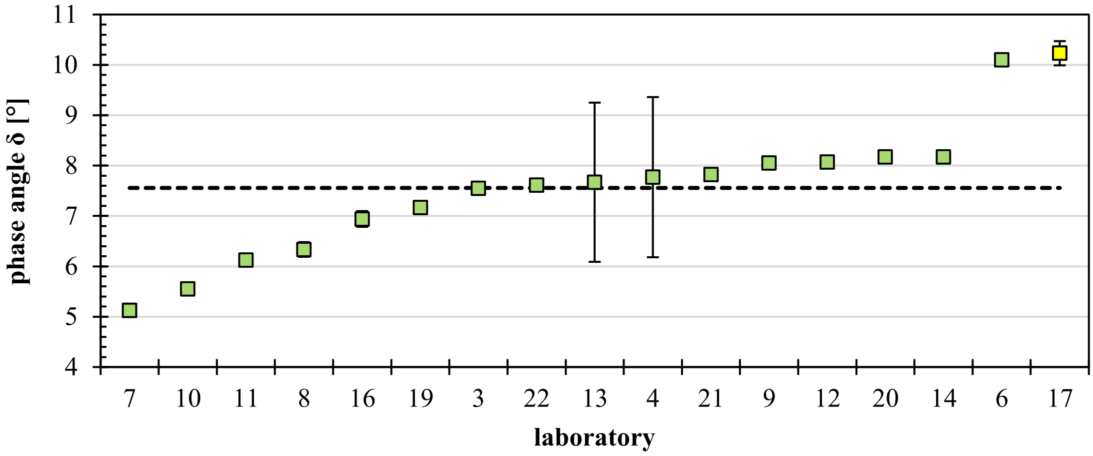

Figure 41: Phase angle δ of plain binder 50/70 at 1.0Hz and -30°C

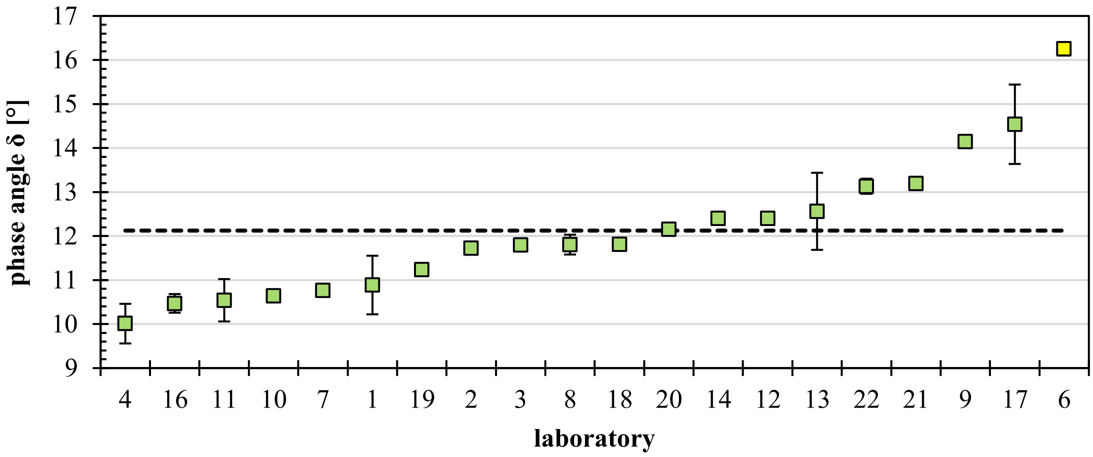

Figure 42: Phase angle δ of plain binder 50/70 at 1.0Hz and -20°C

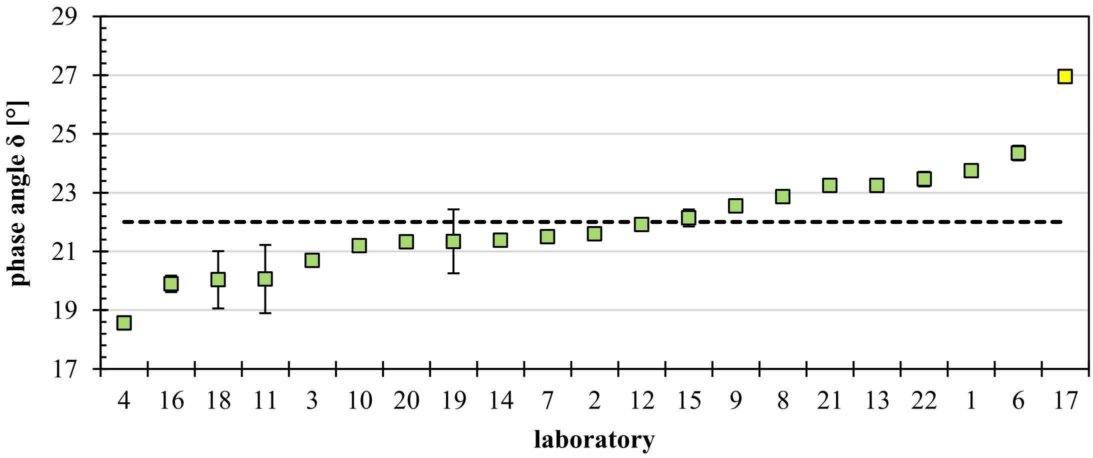

Figure 43: Phase angle δ of plain binder 50/70 at 1.0Hz and -10°C

https://doi.org/10.24355/dbbs.084-202111031047-0

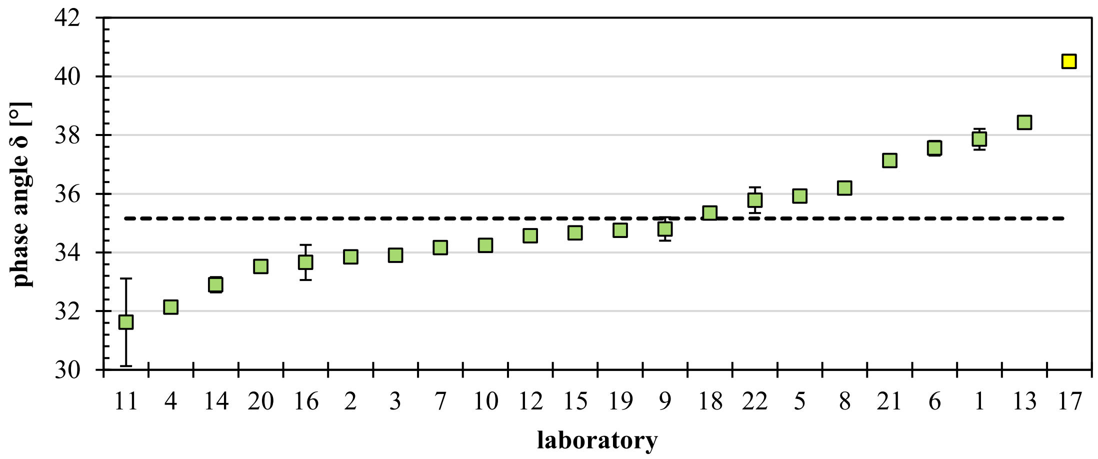

Figure 44: Phase angle δ of plain binder 50/70 at 1.0Hz and 0°C

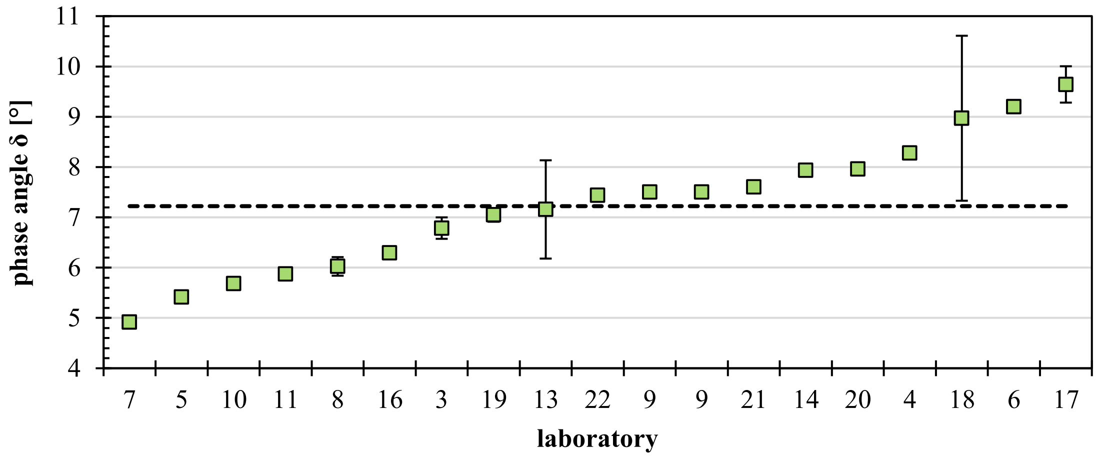

Figure 45: Phase angle δ of polymer modified binder 25/55-55 at 1.0Hz and -30°C

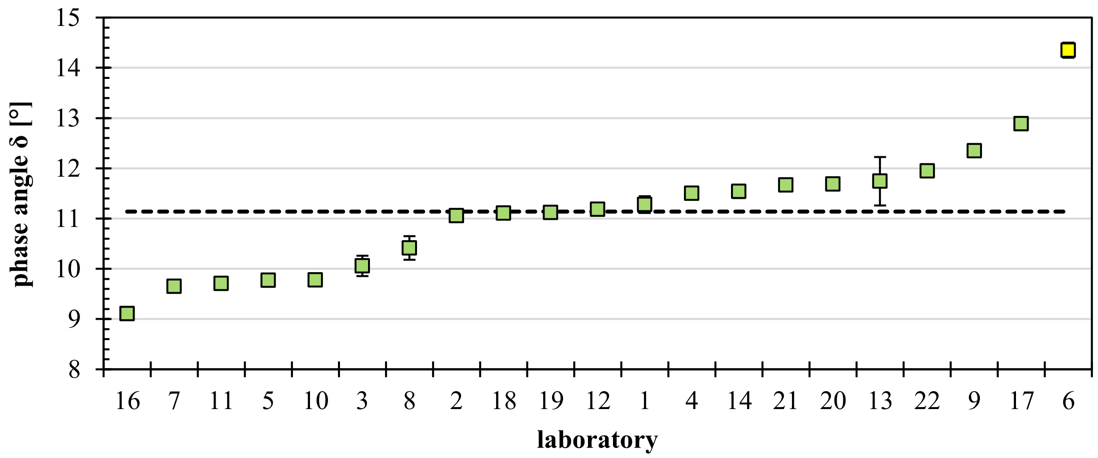

Figure 46: Phase angle δ of polymer modified binder 25/55-55 at 1.0Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

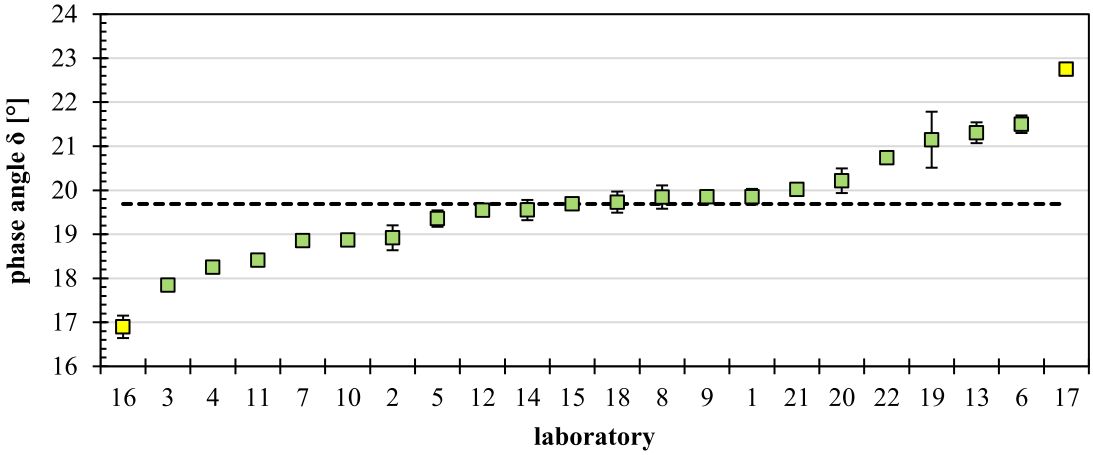

Figure 47: Phase angle δ of polymer modified binder 25/55-55 at 1.0Hz and -10°C

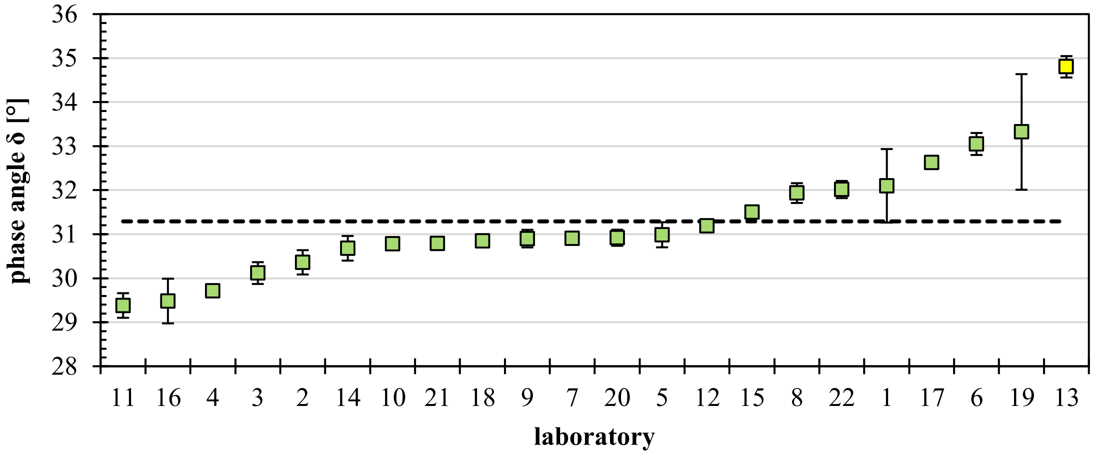

Figure 48: Phase angle δ of polymer modified binder 25/55-55 at 1.0Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.8 Graphical analysis of rheological parameters @10.0Hz

6.8.1 Complex Shear Modulus G*

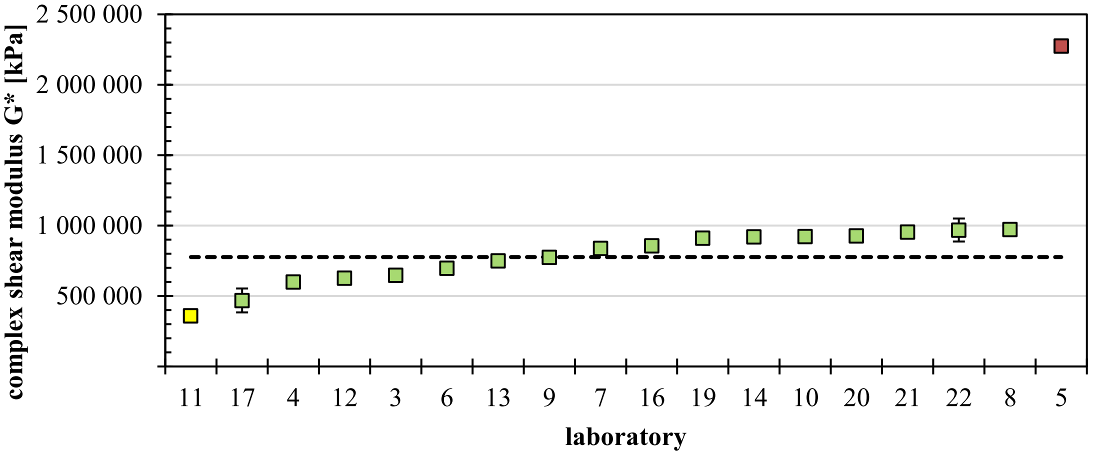

Figure 49: complex shear modulus G* of plain binder 50/70 at 10.0Hz and -30°C

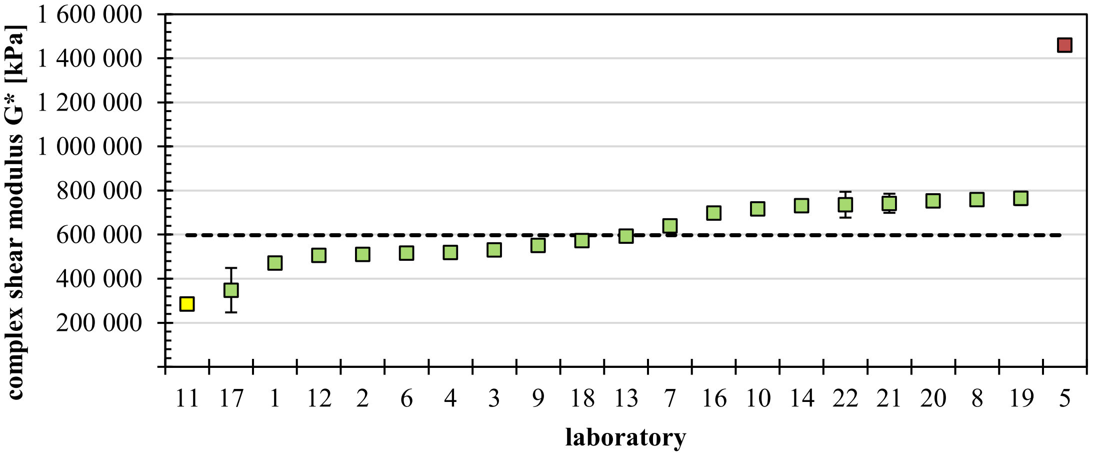

Figure 50: complex shear modulus G* of plain binder 50/70 at 10.0Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

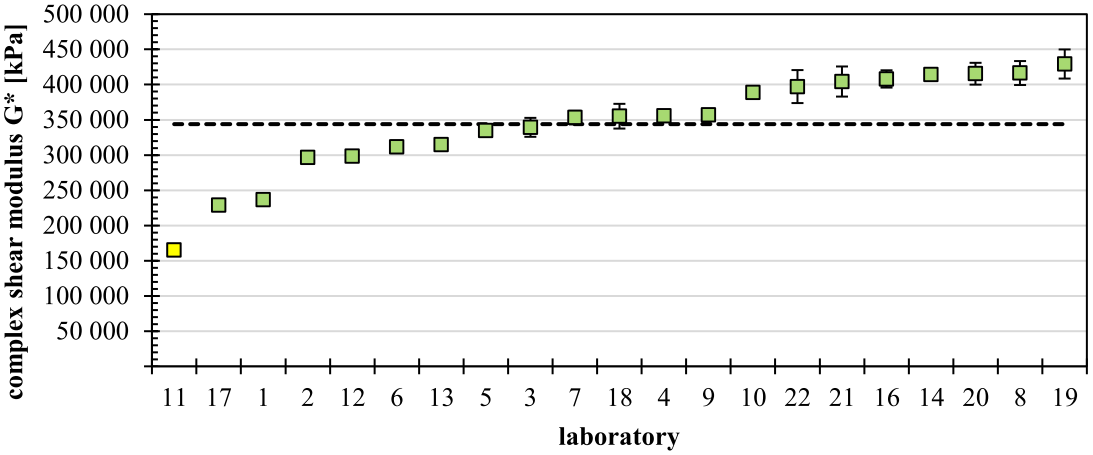

Figure 51: complex shear modulus G* of plain binder 50/70 at 10.0Hz and -10°C

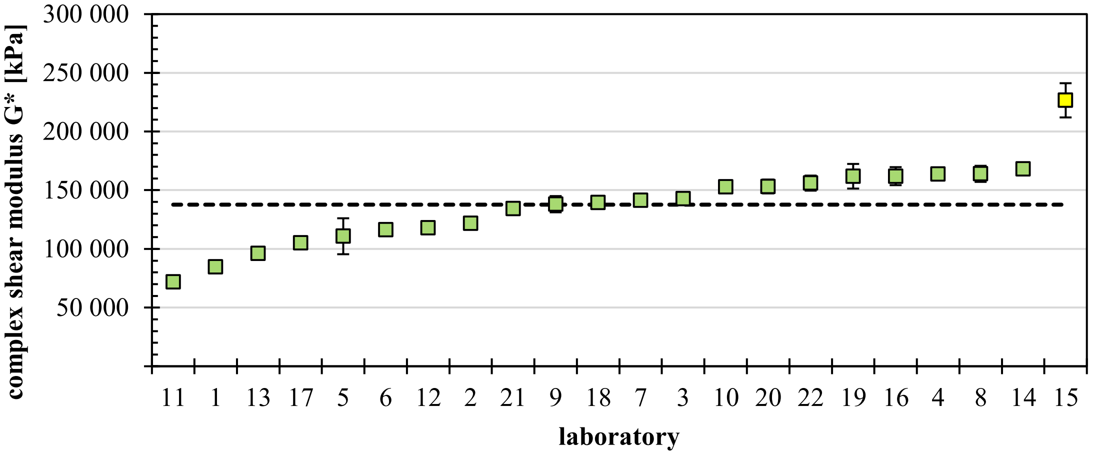

Figure 52: complex shear modulus G* of plain binder 50/70 at 10.0Hz and 0°C

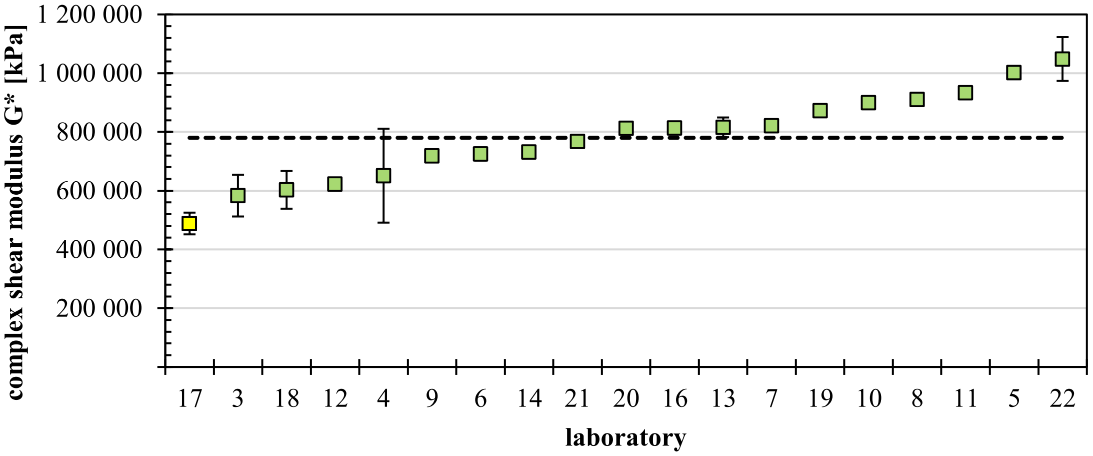

Figure 53: complex shear modulus G* of polymer modified binder 25/55-55 at 10.0Hz and -30°C

https://doi.org/10.24355/dbbs.084-202111031047-0

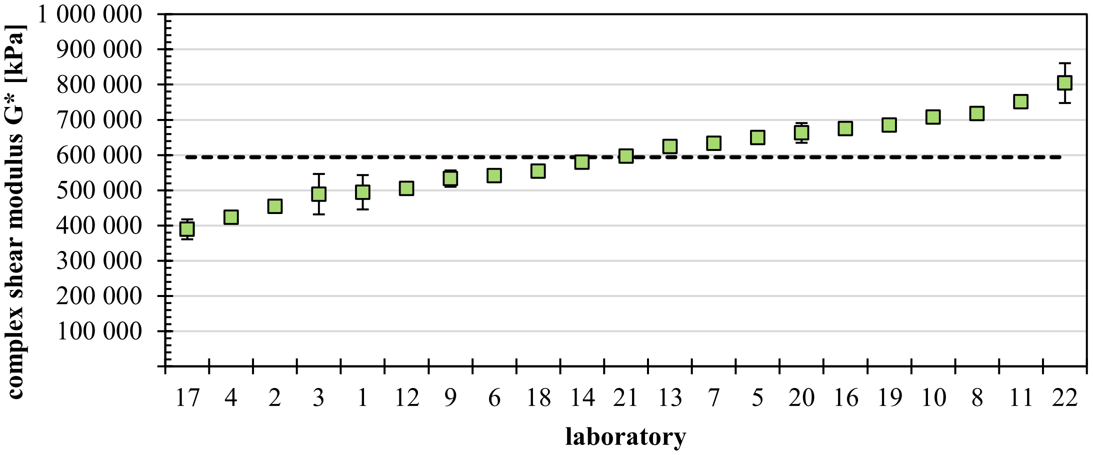

Figure 54: complex shear modulus G* of polymer modified binder 25/55-55 at 10.0Hz and -20°C

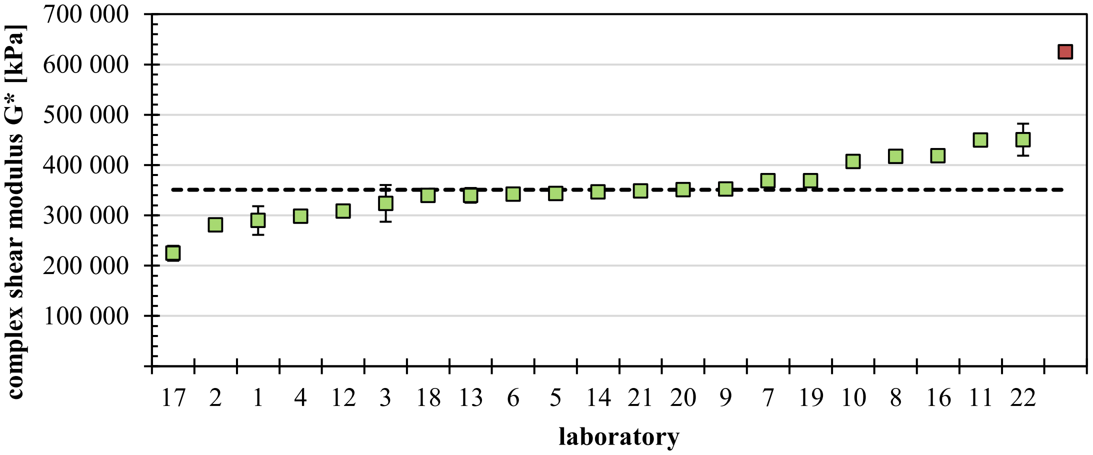

Figure 55: complex shear modulus G* of polymer modified binder 25/55-55 at 10.0Hz and - 10°C

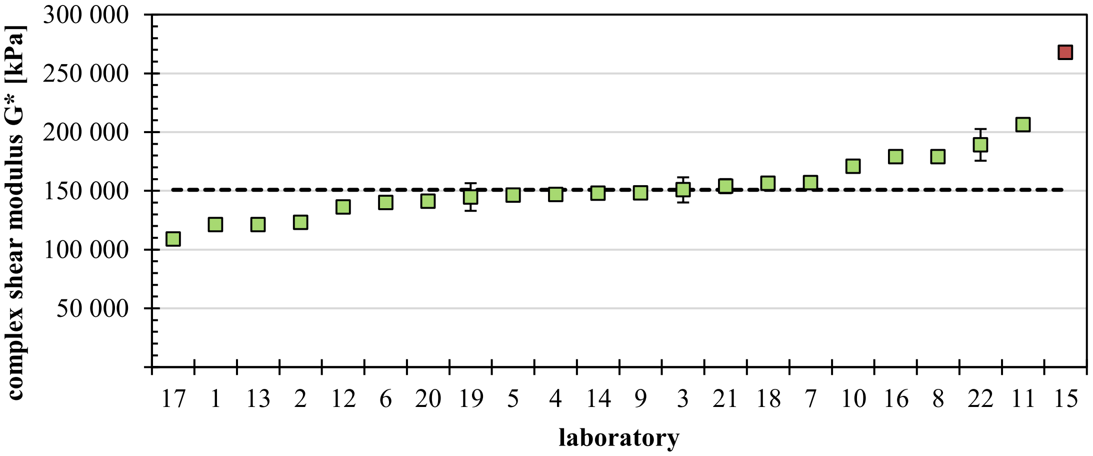

Figure 56: complex shear modulus G* of polymer modified binder 25/55-55 at 10.0Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

6.8.2 Phase angle δ

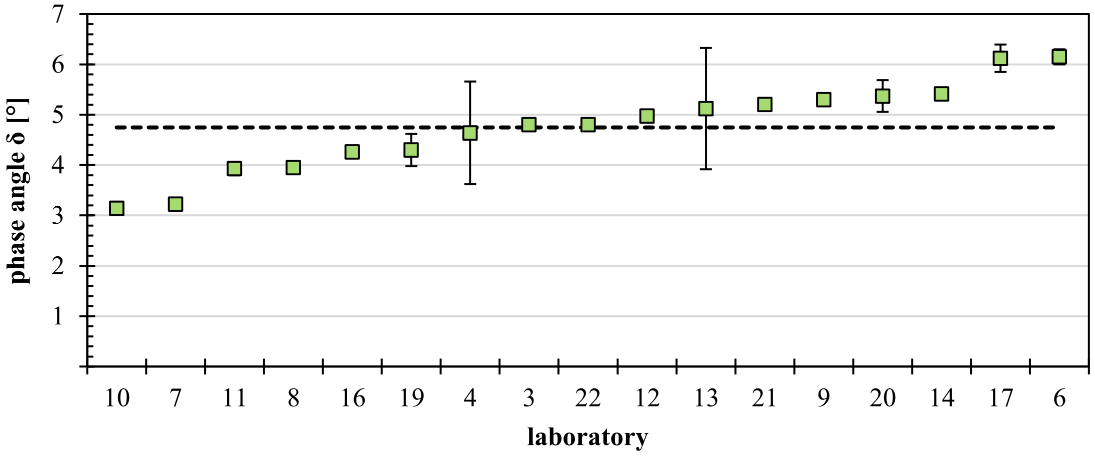

Figure 57: Phase angle δ of plain binder 50/70 at 10.0Hz and -30°C

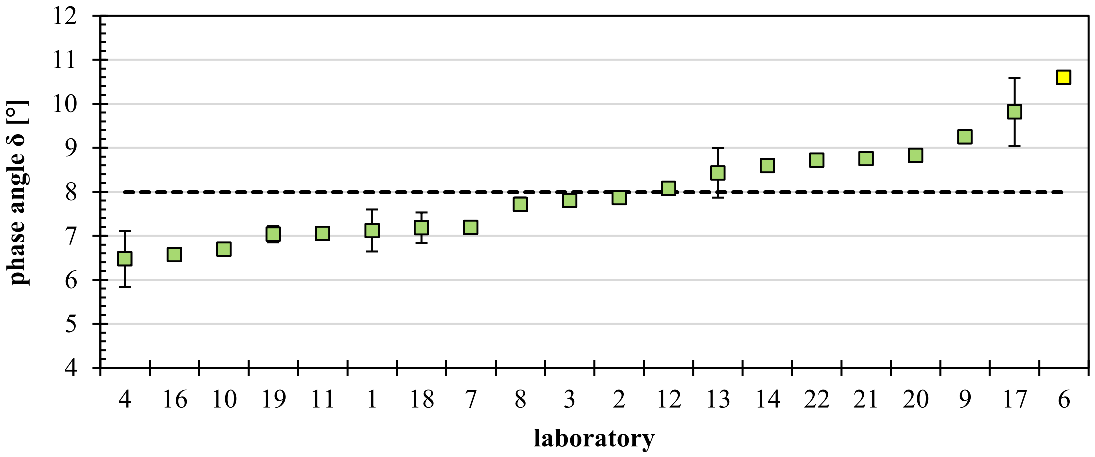

Figure 58: Phase angle δ of plain binder 50/70 at 10.0Hz and -20°C

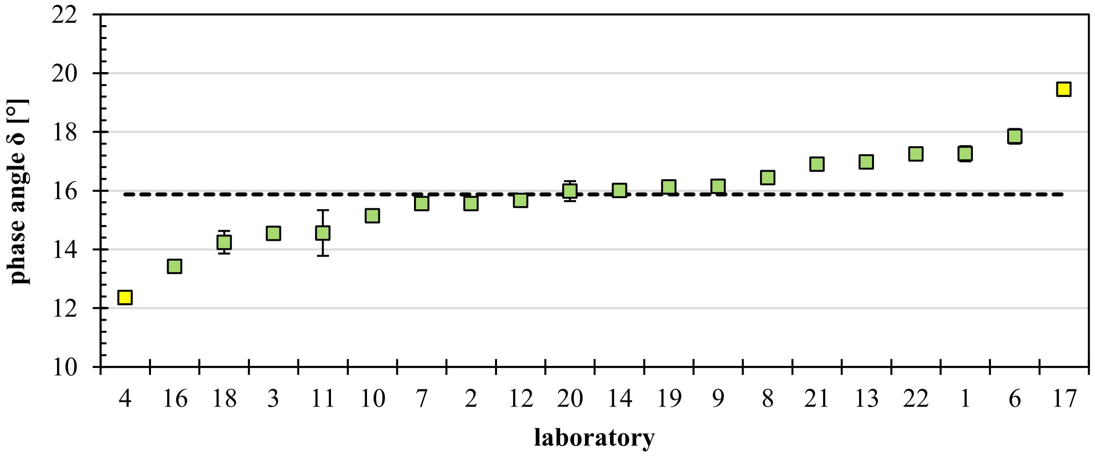

Figure 59: Phase angle δ of plain binder 50/70 at 10.0Hz and -10°C

https://doi.org/10.24355/dbbs.084-202111031047-0

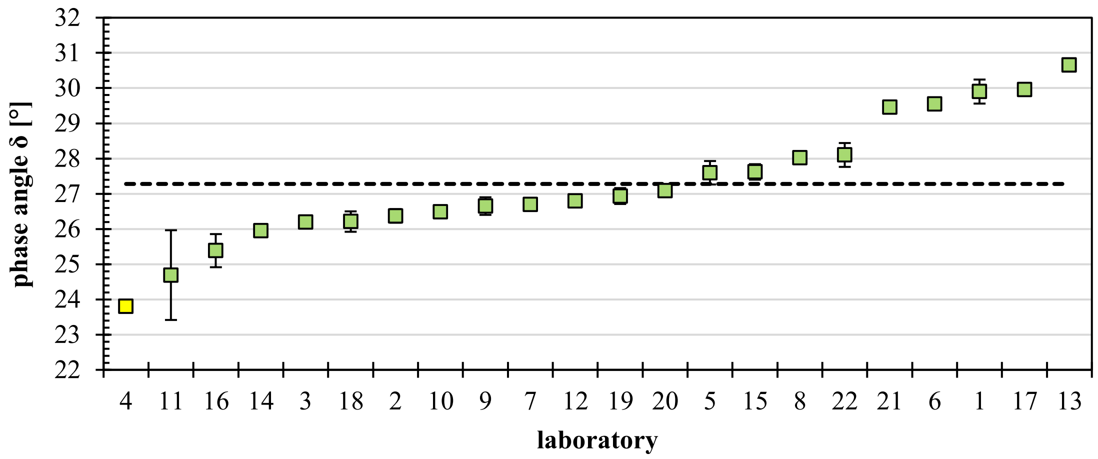

Figure 60: Phase angle δ of plain binder 50/70 at 10.0Hz and 0°C

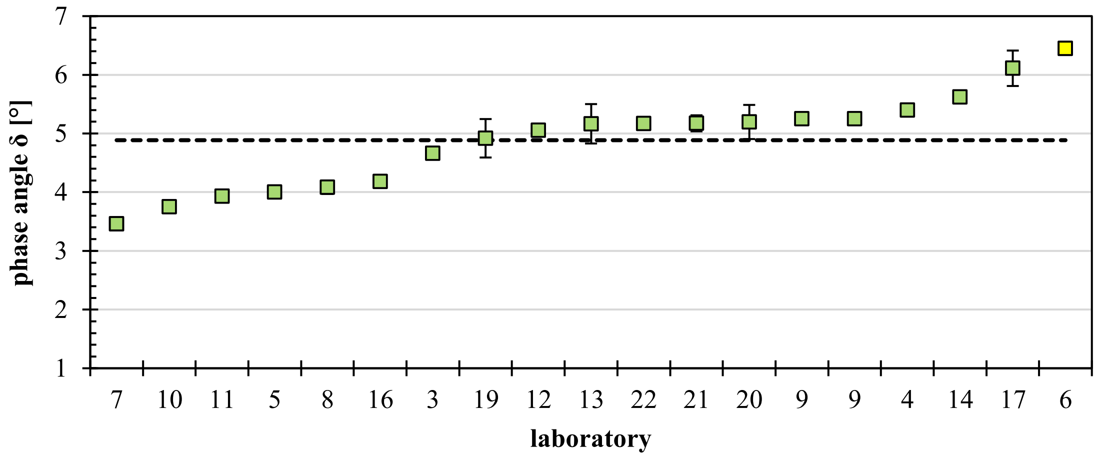

Figure 61: Phase angle δ of polymer modified binder 25/55-55 at 10.0Hz and -30°C

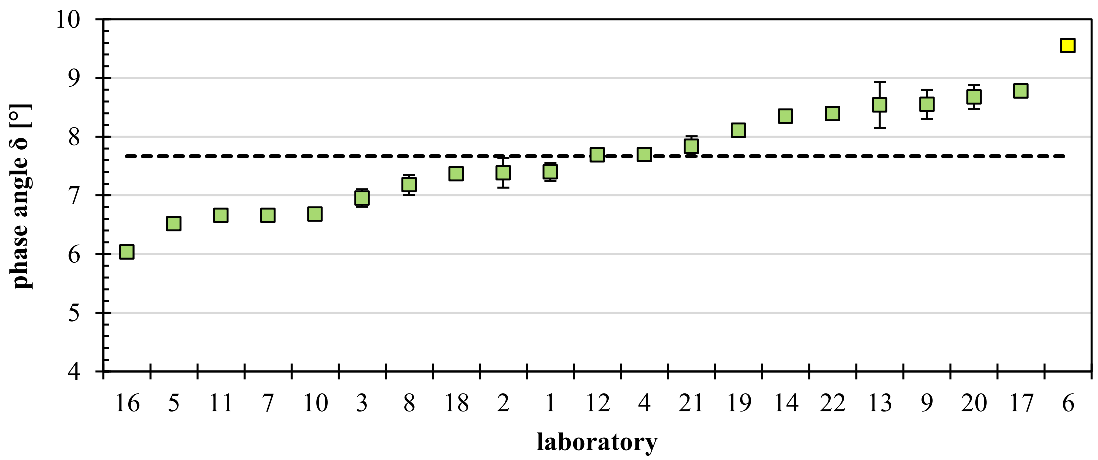

Figure 62: Phase angle δ of polymer modified binder 25/55-55 at 10.0Hz and -20°C

https://doi.org/10.24355/dbbs.084-202111031047-0

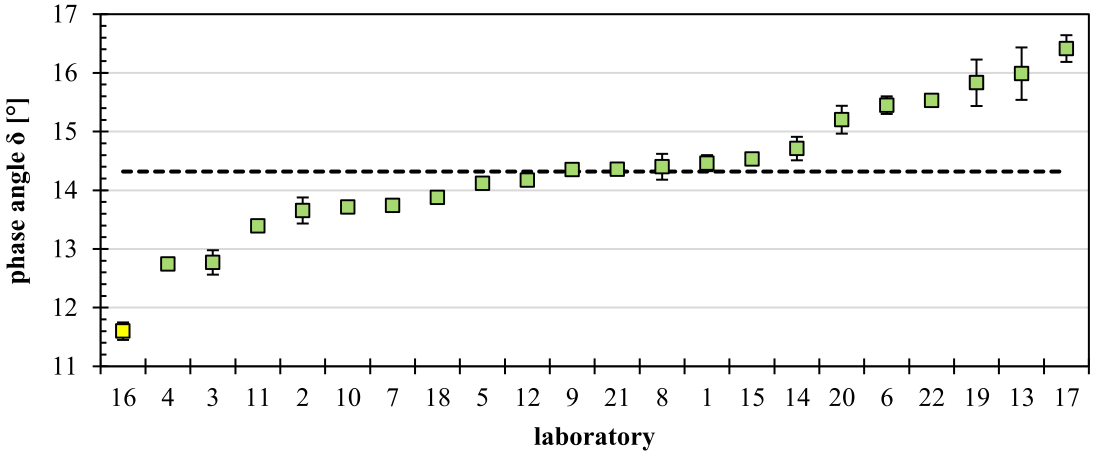

Figure 63: Phase angle δ of polymer modified binder 25/55-55 at 10.0Hz and -10°C

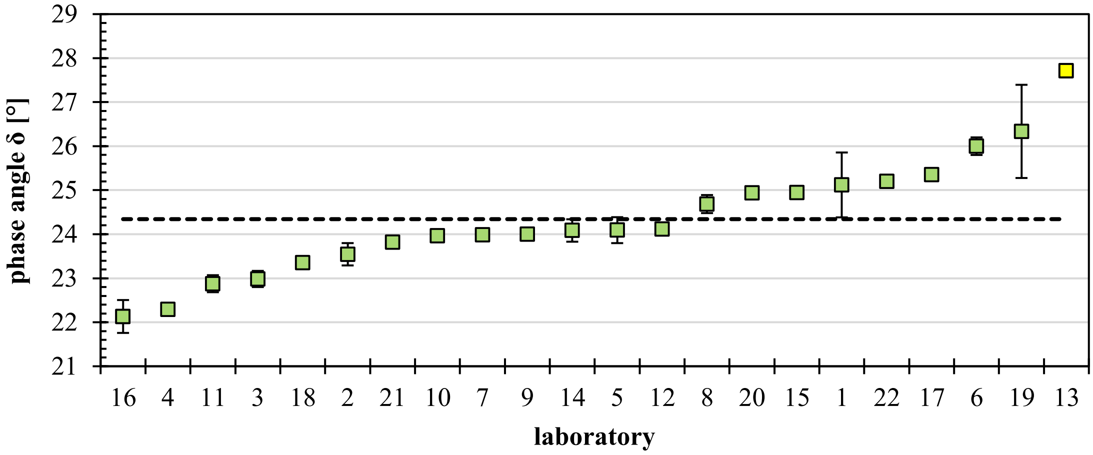

Figure 64: Phase angle δ of polymer modified binder 25/55-55 at 10.0Hz and 0°C

https://doi.org/10.24355/dbbs.084-202111031047-0

7 Summary and conclusions

This interlaboratory study of 20 participants includes DSR test results on rheological assessment of low temperature properties of asphalt binders using the 4 mm parallel plate geometry. Two asphalt binders were considered, plain binder of the type 50/70, and polymer modified asphalt binder of the type 25/55-55 A.

All participants performed temperature-frequency-sweep tests from -30 to 0°C and from 0.1 to 10Hz using their laboratory specific testing protocols.

22 individual test results were received, as one of the laboratories provided three test results with different sample preparation procedures. Along with the test results, information on sample geometry, sample preparation, sample conditioning and test procedure was collected. A limited statistical analysis was performed in order to analyze repeatability and reproducibility of the test results.

The following conclusions can be drawn:

- different Dynamic Shear Rheometer types were used, including equipment from TA Instruments. Malvern (now Netzsch) and Anton Paar;
- varying liquids are in use for counter cooling;
- compliance correction is performed by 10 participants, with compliance values in the range of 0.0014 and 0.028;
- 11 participants had experience with the 4mm geometry already;
- 15 participants use silicone moulds for sample preparation;
- sample loading temperature varies from 25 to 90°C;
- 19 participants used a heated stainless steel trimming tool with an approximate temperature of 90 to 100°C;
- the sample height (gap width) varies between 1 and 3.1mm, and the majority of participants use 1.75mm;
- equilibrium time varies between 2 and 45 minutes;
- the majority of participants uses controlled-strain mode, with strains varying from 0.005 to 0.1%; for controlled-stress mode the stress varies from 10 to 300kPa;
- a wide scattering is observed for both complex shear modulus and phase angle; deviation of individual values for each data set is usually below 10% for complex shear modulus. and below 5% for phase angle;
- some provided results are implausible or inconsistent, which do certainly influence the statistical analysis, however, no systematic dependencies between individually chosen test parameters and test results were determined at this point.

It should be noted, that the test results are obtained from varying test setups, regarding sample preparation, sample geometry, sample conditioning and testing. Thus, the results are not obtained under reproducibility conditions, and they provoke deviations up to 40% from the general mean value for complex shear modulus, and up to 30% for phase angle.

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From this first interlaboratory testing campaign it is concluded that the 4 mm parallel plate geometry in the DSR is generally applicable for testing asphalt binder at low temperature conditions, resulting in acceptable repeatability. However, the test procedures need to be unified and standardized. Therefore, a follow-up interlaboratory study is carried out in 2020 with more detailed instructions regarding sample preparation, sample geometry, sample conditioning and test procedure.

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